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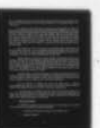
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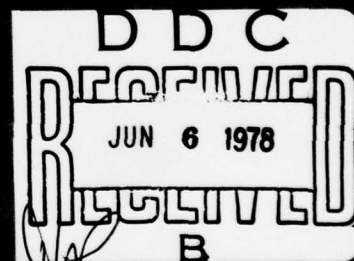


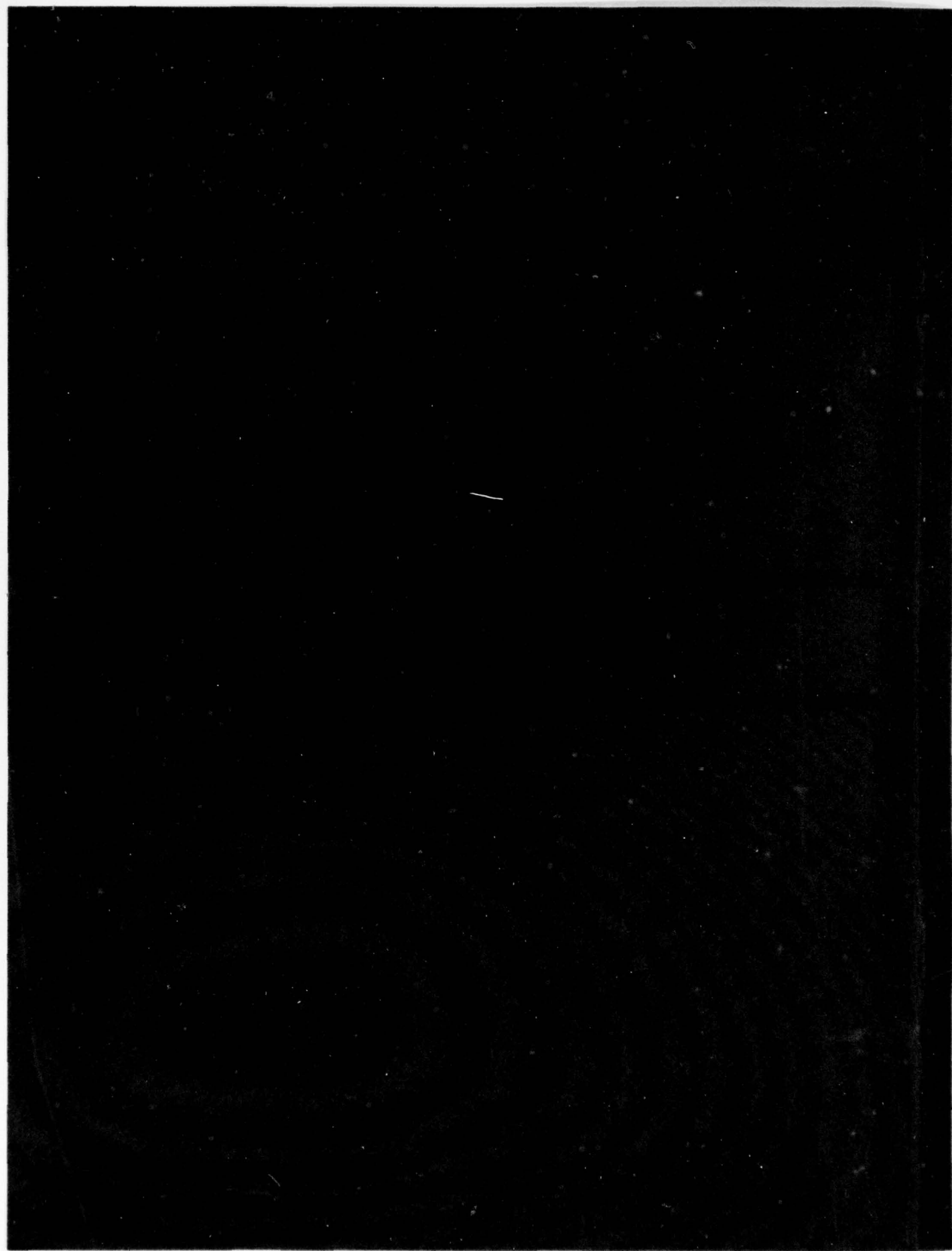


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## PREFACE

The work described in this report was conducted under Demilitarization/Disposal Project 728012.21, Demilitarization of One-Ton Containers of Mustard Agent (HD), at Fort McClellan, Alabama. The active phase of this work was started in January 1974 and completed in September 1975.

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## SUMMARY

In January 1974, work was initiated to dispose of eight one-ton containers of bulk mustard at Fort McClellan, Alabama. The agent was sampled and analyzed. Planning, process development, and design progressed favorably to a point where the design of the system was finalized and fabrication was begun. Programs for testing the operational theory of the unit were also developed and demonstrated at Edgewood Arsenal (now Chemical Systems Laboratory) and Rocky Mountain Arsenal. Coordination with Fort McClellan for operations site setup and with Anniston Army Depot for utilization of operating personnel had progressed satisfactorily as well. By September 1975, both the Basic Plan and the Environmental Impact Assessment for the demilitarization of one-ton containers of mustard agent (HD) at Fort McClellan, Alabama, had reached the Department of the Army for review.

It was subsequently decided that the demilitarization operation would be held in abeyance pending further study of a plan to collocate the subject agent from Fort McClellan to Anniston Army Depot. In light of the Department of the Army decision to halt the project, actions were taken at Edgewood Arsenal to lay away incomplete equipment, to cancel current orders, and to place useful equipment and chemicals in other active demilitarization programs. All work at Fort McClellan was halted and coordination with both Fort McClellan and Anniston Army Depot was ended.

Because the equipment and data generated by the Fort McClellan mustard demilitarization program were largely applicable to other programs, steps were taken to make the maximum use of results outlined in this report. The remaining equipment is being held for possible use in future demilitarization programs as directed by the Department of the Army Project Manager for Chemical Demilitarization and Installation Restoration (CDIR).

The plan to collocate all agent stocks at Fort McClellan was approved by Headquarters, Department of the Army, in November 1976. This move included the eight one-ton containers, some mustard-filled 155mm projectiles and GB-filled 105mm projectiles, and a 20-ml vial of GB. The move was made on 14 December 1976 from Fort McClellan, Anniston, Alabama, to Anniston Army Depot, Bynum, Alabama, without incident. The eight one-ton containers were consolidated as part of the stockpile at Anniston Army Depot.

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# DEMILITARIZATION OF MUSTARD AGENT (HD) AT FORT McCLELLAN, ALABAMA

## I. INTRODUCTION.

This report documents the progress of the mustard demilitarization and disposal program for Fort McClellan, Alabama, from its beginning in January 1974 to July 1976. This includes the active phase of the project which lasted until work was halted in September 1975 and the subsequent passive phase. Work was halted by the Department of the Army pending decisions involving collocation of the subject agent instead of its destruction at Fort McClellan.

## II. BACKGROUND.

### A. Early History of Mustard Agent at Fort McClellan.

There were eight type D one-ton containers (figure A-1, appendix A)\* at Fort McClellan, Alabama, containing varying amounts of distilled mustard agent (HD), as shown in table 1. The containers held a total net weight of 9,765 pounds of HD. Originally, the one-ton containers\*\* were used by the Chemical Center and School at Fort McClellan for instruction in chemical agent transfer. When this function ceased, the agent was scheduled to be sea-dumped during Operation CHASE in 1969. Oil was to be added to each ton container to assure negative buoyancy during the sea-dump. Operation CHASE was subsequently cancelled and no oil was added to the one-ton containers.

Table 1. Levels of Fill for One-Ton Containers at Fort McClellan

Container stencil No.	Container serial No. (or stamped No.)	Mustard net weight*	Mustard purity (minimum)
		lb	%
1	10549**	1,130	87.2
2	78025	990	84.4
3	27328	1,485	88.0
4	14479**	910	87.2
6	085817	990	89.3
7	081151	1,200	88.8
8	28416	1,880	88.8
9	1875	1,180	88.8

\* Total net weight - 9,765 pounds.

\*\* Stamped number.

\* Figures A-1 through A-18 are listed in appendix A.

\*\* One-ton containers are sometimes referred to as "ton containers" throughout this report.

B. First Request for Transfer of Mustard Agent.

In 1972 and 1973, the Chemical Center and School functions were transferred to Redstone Arsenal, Huntsville, Alabama, and to Aberdeen Proving Ground, Maryland. Authority was requested by Fort McClellan to transfer the ton containers to nearby Anniston Army Depot, Bynum, Alabama, which has a storage capability. This request was denied by the Department of the Army and in late 1973 the Army Materiel Command (AMC) Program Manager for Demilitarization of Chemical Materiel was requested to arrange for onsite detoxification and disposal of the material at Fort McClellan.

C. Tasking for Onsite Detoxification.

In January 1974, the Demilitarization/Disposal Office and, in turn, the Manufacturing Technology Directorate at Edgewood Arsenal were tasked by the AMC Program Manager for Demilitarization of Chemical Materiel with the mission of detoxifying and ultimately disposing of the eight one-ton containers at Fort McClellan.

III. CONCEPT OF THE MUSTARD DEMILITARIZATION OPERATIONS.

A. Detoxification Options.

The concept of the mustard demilitarization operations was developed during the time frame of January 1974 to December 1974. The overall scheme of the operation was achieved by evaluating several options: incineration, chemical neutralization, and transportation to a storage depot. Incineration was eventually ruled out as being too costly; transportation was ruled out, at the time, because of then-current Department of the Army direction. Several methods of chemical neutralization were advocated. Monoethanolamine (MEA) was chosen as the decontaminant in preference to sodium hydroxide (NaOH) and to calcium hypochlorite (HTH) based on the stringent criteria established for the decontamination of the system.

B. Choice of Decontaminant.

The criteria for decontamination of the system as of January 1974 dictated that the selected reaction process must decontaminate the mustard under the following conditions:

1. The reaction process must not allow more than  $0.003 \text{ mg/m}^3$  to escape into the air in vapor form.
2. The reaction process must be efficient enough to reduce the concentration of mustard to less than 5 ppm in the liquid waste [later reduced to 2 ppm in review with the Department of Health, Education, and Welfare (HEW)].
3. The resulting waste residue must pass Department of Transportation (DOT) tests as a class B poison (or less) for transportation to its ultimate disposal location.
4. Sufficient pilot data must be available on both the reaction process and monitoring methods to be acceptable to all reviewing agencies.

5. A single-phase solution would be desirable from both chemical reaction and physical handling aspects.

Monoethanolamine was finally chosen over NaOH and HTH for two major reasons. First, it was the decontaminant recommended by Edgewood Arsenal's Chemical Laboratory for use with mustard (see appendix B). Second, current research into the MEA-mustard reaction insured that all five criteria listed above would be met within the year, although knowledge gaps for the remaining two decontaminants were not currently being investigated.

#### C. Reactor Size.

The reactor size was the next critical step in the building of the operational concept as the reactor vessel was to be the heart of the demilitarization unit. The choice of a reactor was based on the following criteria:

1. Operations were to be of as short duration as possible to minimize costs.
2. The unit was to be fabricated offsite and transported to the disposal site at Fort McClellan.
3. The reactor materials of construction were to be compatible with the MEA-mustard reaction, and the configuration was to be scaled to the pilot batch of 55 gallons.

At first, a 55-gallon reactor or a bank of such reactors was considered. For the volume of agent involved, a larger reactor was deemed desirable. This was mainly because the heat load of the reaction severely limited the amount of mustard that could be fed to a small reactor.

Next, a 2000-gallon reactor was investigated; however, this was discarded due to the transportability problem. The compromise reactor chosen was a 500-gallon unit made of type 316 stainless steel.

#### D. Transfer and Containment.

In the designing of the overall system, the criteria for transferring and decontaminating mustard under total containment (to  $0.003 \text{ mg/m}^3$ ) were of overriding concern. To achieve this goal, the system was to be totally enclosed by a cabinet that could contain any spillage from either the one-ton container, the reactor, or the transfer system between them. Due to the unwieldy size of such a cabinet, the unit was ultimately designed as two cabinets, a reactor cabinet and a one-ton container cabinet, with a track and trolley for moving the bulky container in and out of the ton container module (figure A-2). The two cabinets would be connected when assembled at the disposal site.

The transfer system was a series of stainless-steel pipe lines to carry both decontaminant and agent to the reactor and to carry reacted products away from the reactor. Attached to the transfer system were various pumps and holding tanks.

To charge the system with MEA, a 10-gal/min positive-displacement pump was chosen. This pump was to be powered by a class 1, group D, explosion-proof motor. The pump



was to be located in the work area at the front of the cabinet and was to be attached to the system with a flexible rubber hose. From there the piping was to run through the transfer tank to the reactor.

To charge the system with mustard, a vacuum pump was provided. The pump was piped behind two series-connected charcoal particulate filters; the discharge side of the pump facing the atmosphere, the suction side of the pump facing the filters. The piping, after leaving the filters, entered the cabinet, and ran upward and through a ball valve to the transfer tank. Thus a vacuum could be pulled on the transfer tank. The tank itself was connected by stainless steel piping and flexible rubber hose to the ton container. Thus the vacuum from the transfer tank could be transmitted to the ton container. The ton container was to be vented to the reactor vent line, which exited the cabinet via a surge tank and two series-connected charcoal particulate filters.

The transfer tank was to be the center of the transfer system. After mustard had been transferred to this tank, it was to be charged to the reactor by gravity. This could be done by venting to the reactor vent line and opening a one-inch ball valve between it and the reactor. A sight glass on the tank was to allow visual inspection of the fill level.

Vapors exiting the reaction were to leave through a six-inch vent line in the top of the reactor. This line was to pass through a condenser and then down through a 55-gallon surge tank. The line then was to exit the surge tank and the cabinet and was to vent into two series-connected charcoal particulate filters. Neither this system nor the vacuum system described in the preceding paragraphs was to use motor-driven filters to draw air. Air was simply to be filtered as it was supplied by the internal pressure differences in the system caused by liquid level changes, reaction vapor pressures, or venting.

Monoethanolamine at elevated temperatures is considered a moderate fire hazard. As a safety precaution, a nitrogen purge was to be added to flow through the transfer tank and into the reactor. This flow could be operated in either a purge or flood mode to preclude air entry and any possibility of fire or explosion in case of reaction overtemperature.

For the purpose of dumping the reactor and surge tank, a 20-gal/min positive-displacement pump was to be provided. This was to be connected by flexible tubing and two-inch (OD) stainless steel pipe to the bottom of the reactor and surge tank.

A small line was to be attached to the side of the reactor to allow sampling of the batch. This was routed through two spring-shut-type valves, one inside the cabinet and one outside. Also, a piping system was to be connected to provide a deluge wash of the cabinets with MEA in case of internal contamination of the cabinet.

#### E. Cabinet and Ventilation.

As described in section III, the entire process was to be surrounded by two cabinets. These cabinets were to be designed to provide the following:

1. Containment of the agent and vapors in case of spills or leaks.
2. Adequate ventilation.

3. Access by repair personnel.
4. Draining for decontaminant or water.
5. Adequate lighting.
6. Ability to easily insert or retract one-ton containers.

Ventilation was to be provided by connecting two M6A2 electric-blower-driven charcoal filter units to the reactor cabinet. These units would draw air from the smaller one-ton container cabinet where an antibackdraft valve was located in the rear. This valve was to be a weighted valve designed to close in the event of filter blower failure. The system was designed to maintain at least 0.5 inch of water vacuum within the cabinets at all times when operational. A fan for circulating air was to be provided in the aerodynamic dead spot where the cabinets met. This fan was to be powered by an explosion-proof motor.

Access to the cabinet components could be gained by three entrances. The first and easiest was through the sliding door of the one-ton container cabinet. This provided access to the transfer piping, circulation fan, and all components in the one-ton container area. The second entrance was to be a bolted door, on the left side of the reactor cabinet, that gave access to the surge tank area of the reactor cabinet. The third entrance was to be a bolted door on the top of the reactor cabinet. This entrance was to be provided for servicing of the thermocouples, the agitator, and the transfer piping that were not accessible from the one-ton container cabinet.

Cabinet drainage was to be provided by placing a floor drain in the left end of the reactor cabinet. This floor drain could be used for both cabinets as the unit was to be constructed with the floors sloped such that all liquid would flow to the drain.

Lighting was to be provided from two sources. A four-tube fluorescent fixture was to be centered over the top access door in the reactor cabinet to provide lighting in that cabinet. A two-tube fluorescent unit centered over the face of the one-ton container was to provide lighting for the cabinet.

The one-ton container was to be placed on a manually-pushed trolley and passed into the cabinet through a sliding door that was to be gasketed so a tight seal would be maintained during operations.

#### F. Overall Operational Concept.

Step-by-step operational procedures were developed for the use of the demilitarization equipment. A brief outline of these is included in appendix C. The following is envisioned as the scenario for the demilitarization of the one-ton containers.

The one-ton container was to be removed from the thaw tent where it was to be kept at a temperature of approximately 70°F to insure that the contents remained liquid. By use of heavy-duty forklift, the one-ton container was to be lifted onto the one-ton container trolley and inserted into the one-ton container cabinet. After sealing the cabinet, the one-ton container was to be weighed (using the transducer system provided) and then was to be connected to the transfer piping by the use of the flexible hose and quick-disconnects available. This was to be

accomplished by a single worker operating through the glove ports provided in the one-ton container cabinet. The worker also was to open the one-ton container valves.

The next action was to prefill the reactor with 200 gallons of MEA. Using the transfer system, the operators were then to apply a vacuum in the transfer tank and, in turn, apply that vacuum on the one-ton container. In this manner, 15 gallons of mustard were to be transferred to the transfer tank as observed on the sight glass.

After the agitator, the temperature monitors, and the condenser cooling water were started, the mustard was to be slowly charged to the reactor. The transfer tank then was to be flushed with 25 gallons of MEA which, in turn, was to be fed to the reactor. The temperature of the reaction was to be monitored and after completion of the reaction the batch was to be cooled by turning on the reactor jacket cooling water. Completion of the reaction was to be determined by actual laboratory analysis of reactor samples prior to dumping the reactor. For safety purposes, the jacket could also be used during a reaction if excess temperatures were detected. A nitrogen purge to eliminate any chance of explosion also was to be provided as a standby emergency system.

After completing one incremental batch of 15 gallons of mustard and 25 gallons of MEA, this process was to be repeated. The process could be run four times per initial batch of 200 gallons of MEA. This would correspond to a final batch of 60 gallons of mustard and 300 gallons of MEA. The rationale for this was the idea of using the 5:1 MEA-mustard decontamination-volume ratio recommended by the Edgewood Arsenal Chemical Laboratory and pilot studies, although never using less than the 10:1 MEA-mustard volume ratio that dictates safe reaction temperatures (see section IV.B). Consequently, per incremental batch, the ratio of MEA to unreacted mustard was never to be less than 10:1, and the final decontamination ratio was to result in a 5:1 ratio. After four increments, the batch was to be sampled, checked for residual contamination, and then emptied into 55-gallon drums. The process was to be repeated until the one-ton container was emptied. The overall time involved for decontaminating one batch of 60 gallons of mustard was estimated to take one 40-hour week from setup time through drumming of the waste liquid.

After emptying the one-ton container of all mustard possible, the transfer system was to be used to charge the one-ton container with 100 gallons of MEA. This was to be accomplished in two 50-gallon batches. Again temperature was to be closely monitored. After samples were checked for residual contamination, the one-ton container was to be sealed, removed, and stored with the waste liquid drums. The next one-ton container then was to begin the cycle of decontamination.

Ultimately, after all one-ton containers were decontaminated, the equipment was to be laid away at Anniston Army Depot for possible future use. The waste liquid in both 55-gallon drums and one-ton containers was to be sent to Rocky Mountain Arsenal (RMA), Denver, Colorado. Once at RMA, the waste liquid was to be incinerated, using the incineration plant available at that location. The resulting solid, inert salt waste, collected as a result of scrubbing the furnace gases, was to be stored with similar mustard salt wastes at RMA awaiting disposition. All 55-gallon drums and one-ton containers were to be disposed of as scrap iron.

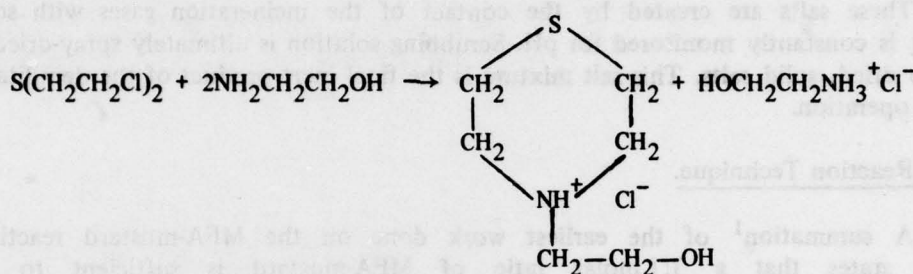


#### IV. DETOXIFICATION REACTION AND TECHNIQUE.

##### A. Reaction Chemistry.

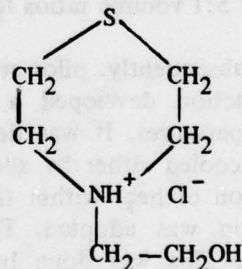
The chemical decontamination of the mustard was accomplished by mixing it with MEA. The reaction of these two liquids results in a homogeneous waste liquid mixture. The reaction products were not analyzed to give relative percents of each component because this was not necessary to the operation. The mixture passed Department of Transportation (DOT) screens assuring that it was less than a class B poison (see appendix D). Due to the presence of excess MEA, the liquid had to be labeled as corrosive and was subsequently classified as "corrosive, not otherwise specified (NOS)" by the Department of Transportation.

The major equation to describe the reaction is as follows:

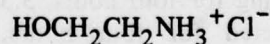


This results in the following final products:

1. N-(2-hydroxyethyl)-thiomorpholine hydrochloride:

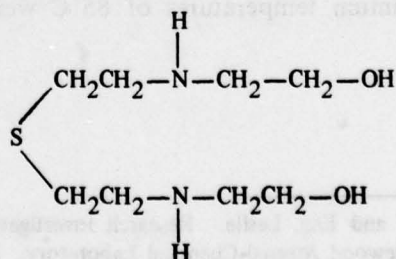


2. Monoethanolamine hydrochloride:



3. Monoethanolamine:  $\text{NH}_2\text{CH}_2\text{CH}_2\text{OH}$

4. Bis(hydroxyethylaminoethyl)sulfide :



The bis(hydroxyethylaminoethyl)sulfide is produced only in very small amounts as a reaction alternate product.

Subsequent incineration of these compounds and scrubbing of the gases with sodium hydroxide (18% NaOH in water) result in the following salts:

1. Sodium carbonate ( $\text{Na}_2\text{CO}_3$ )
2. Sodium chloride ( $\text{NaCl}$ )
3. Sodium sulfate ( $\text{Na}_2\text{SO}_4$ )
4. Sodium sulfite ( $\text{Na}_2\text{SO}_3$ ).

These salts are created by the contact of the incineration gases with scrubbing solution that is constantly monitored for pH. Scrubbing solution is ultimately spray-dried, which produces the dried, solid salts. This salt mixture is the final inert product of the demilitarization and disposal operation.

#### B. Reaction Technique.

A summation<sup>1</sup> of the earliest work done on the MEA-mustard reaction (see appendix B) states that a 3:1 molar ratio of MEA-mustard is sufficient to provide decontamination. A 5:1 volume ratio was chosen therein because it provided a 10:1 molar ratio – the minimum ratio normally accepted for the first-order kinetics desired – and a 230% minimum molar excess of MEA. The early studies, therefore, were completed in the context of the kinetics of 5:1 volume ratios for MEA-mustard.

Subsequently, pilot studies based on the current laboratory data were initiated. The first pilot reaction developed a problem in that the reaction quickly exceeded reasonable operating temperatures. It was determined that, to control the reaction, the process materials needed to be cooled either by added mechanical means or by increasing the reactant volume to allow dissipation of heat within the mix. The method of increasing the reactant volume to allow heat dissipation was adopted. To achieve this, the reaction volume ratio was increased to 10:1 MEA-HD. This was done by cutting the batch volume of mustard in half within each reaction period. Thus, instead of feeding seven gallons of mustard to 35 gallons of MEA in 20 minutes and reacting for four hours, 3.5 gallons of mustard were fed to 35 gallons of MEA in 20 minutes and reacted for four hours, with a second batch of 3.5 gallons of mustard subsequently added to the same reaction mix (now 38.5 gallons) in 20 minutes and allowed to react for an additional four hours. The latter method results in a minimum 10:1 volume ratio of MEA/HD with an ultimate 5:1 ratio to assure optimum safe use of the decontaminant. During this test, maximum temperatures of 85°C were achieved. Results of this test are provided in appendix E.

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<sup>1</sup>Pistritto, Joe, and Eng, Leslie. Research Investigations Concerning the Decontamination of H/HD in Toxic Gas Sets. Edgewood Arsenal Chemical Laboratory. 28 March 1974.



### C. Scaleup for Fort McClellan.

The total agent available at Fort McClellan for detoxification consisted of 925 gallons. The original proposal to dispose of the agent by means of a 55-gallon drum was considered first. In order to meet the safe operating temperature of less than 80°C, specified by the AMC Safety Office, the quantity to be demilitarized would have had to be reduced to less than seven gallons per day. Combined with set-up and tear-down of the unit, it is estimated that only about 21 gallons per week could be effectively detoxified by this technique. Moreover, with all the necessary controls to such a reaction, it was felt that a larger system could be achieved for nearly the same cost.

The final decision, as previously described, was to move to a 500-gallon reactor. An extremely conservative scale-up of the system was used, limiting the maximum temperature to 70°C. The calculations show that, at this point 15 gallons of mustard per day could be detoxified. If cooling of the reaction mix after completion of the reaction had proved adequately fast to permit two increments a day, 30 gallons per day could be detoxified. This latter condition was never verified in test. Assuming the conservative figure of 15 gallons per day, with four operating days per week for reacting and one day for reactor filling, draining, and maintenance, a batch of at least 60 gallons per week would be achieved. This would assure operational completion in four months.

The calculations leading to the figure of 15-gallons-per-increment assumption are shown as follows:

1. The reaction of MEA and HD generates 730 Btu per pound of mustard fed (from the range of 30° to 80°C). This was determined from pilot data.

$$(730 \text{ Btu/lb}) (252 \text{ cal/Btu}) / (454 \text{ gm/lb}) = 405.2 \text{ cal/gm}$$

2. The weight of 15 gallons of mustard is given by:

$$(15 \text{ gal}) (10.6 \text{ lb/gal}) (454 \text{ gm/lb}) = 72,186 \text{ grams}$$

3. Using the formula  $Q = mC_p\Delta T$  and using only the volume and heat capacity of the MEA as a heat sink at the minimum volume point (200 gallons), the following calculations held:

$$C_p \text{ of MEA} = 0.816 \text{ cal/gm}^\circ\text{C}$$

$$\text{Weight of MEA} = (200 \text{ gal}) (8.49 \text{ lb/gal}) (454 \text{ gm/lb})$$

$$= 770,892 \text{ grams}$$

$$(405.2) (72,186) = (770,892) (0.816) (T - 22.5)$$

The final reaction temperature  $T = 69^\circ\text{C}$  when the above equation is solved.

The final scale-up to Fort McClellan quantities was to be by feeding a 15-gallon increment of mustard to a 200-gallon pre-fill of MEA, followed by a 25-gallon flush of MEA.

This was to be followed by three additional increments of 15 gallons of mustard and 25-gallon flushes of MEA to yield a weekly batch of 60 gallons of mustard to 300 gallons of MEA. This preserves the 10:1-or-better volume ratio necessary as noted in section IV. B, simultaneously holding the final reaction-mixture volume ratio of 5:1, duplicating the pilot waste generated previously.

## V. EQUIPMENT DEVELOPMENT.

### A. Final Design.

Prior to freezing the unit design, documentation involving the planned demilitarization operations was sent to all elements of Edgewood Arsenal and the Office of the AMC Program Manager for Demilitarization of Chemical Materiel. Plans were also briefed to the AMC Safety Office, the Department of Defense Explosives Safety Board, the Department of Health, Education, and Welfare, and the Army Environmental Hygiene Agency. A fault tree hazard analysis was conducted as recommended by the AMC Safety Office and several changes recommended as a result of this study were made to the system. After this period of review, the design was frozen and procurement and fabrication were initiated.

### B. Procurement.

Procurement for the demilitarization project was divided into three categories:

1. Procurement for equipment fabrication
2. Procurement for testing
3. Procurement for field operations

Procurement of items for equipment fabrication and testing was begun in December 1974 prior to the project review. Procurement of items for equipment fabrication and testing developed during the review, however, continued through June 1975.

For items that involved the planned field operations, such as analytical chemicals and laboratory equipment, procurement was not initiated until July 1975 and was in progress when the September 1975 project halt occurred. Items that were actually procured and the disposition of the items are discussed in section VIII.

### C. Fabrication.

Preliminary work to make the reactor suitable for the demilitarization operation was executed during December 1974 and January 1975. This consisted of installing a baffled jacket on the reactor, installing interior reactor baffles, and leak testing the reactor and the jacket. Work was then deferred for two months while the project review continued. In April 1975, project review was considered to be far enough along to permit major fabrication to begin.

The first unit to be worked on was the one-ton container module with the accompanying stand and trolley. This unit progressed slowly because of higher priority projects that occupied the same shop; however, by early August 1975, this unit was substantially complete.

Fabrication of the reactor cabinet and the attendant process piping began in July 1975 and by September 1975 had reached about 50% completion. The transfer tank had been fabricated and all process piping put in place. The surge tank had also been fabricated. The cabinet had been fabricated and was awaiting installation of the reactor and process piping when the project was halted. Photographs of the fabrication, to the extent that it was carried, are provided in appendix A (figures A-3 through A-13).

## VI. TEST PROGRAMS.

### A. Pilot Program.

The pilot program for the Fort McClellan project was actually conducted as part of the research into the destruction of obsolete war gas identification (GID) sets. The original test, GID No. 1, was based on Edgewood Arsenal Chemical Laboratory data recommending a 5:1 MEA-HD volume ratio. As described in section IV.B, this resulted in a situation where excess heat was generated.

The second pilot run, GID No. 2, was a modification of the first. Instead of adding seven gallons of mustard to 35 gallons of MEA in one batch, the seven gallons of mustard were fed in two increments. First, 3.5 gallons of mustard were added and allowed to react to maximum temperature. This mixture was then cooled and the second 3.5 gallons were added. This procedure worked well, resulting in a detoxified, DOT-approved reaction product within safe temperatures. Results are shown in appendix E.

A third pilot run was made to confirm the findings of GID No. 2. This run was identified as GID No. 4 and produced virtually identical results as GID No. 2.

A fourth pilot run was made later in the study of the GID sets studies. This run was made with Levenstein mustard (H) instead of distilled mustard (HD) and was identified as GID No. 18. This run produced a similar curve peaking at slightly lower temperatures (approximately 8°C lower).

The data generated by GID No. 2, GID No. 4, and GID No. 18 are the basis for the Fort McClellan system, as described in section III.

### B. Waste Incineration Program.

The waste incineration test program was initiated to demonstrate that the reaction product waste could be disposed of in an existing Government incinerator complex. This testing was accomplished in the period October 1974 to December 1974 at Edgewood Arsenal and Rocky Mountain Arsenal (RMA).

First, small-scale incineration tests were conducted in the Chemical Laboratory at Edgewood Arsenal. The purpose of these tests was to see what the final residue consisted of and how much  $\text{NO}_x$  was given off. At the completion of these tests, it was shown that the final product was similar to that generated during the mustard incineration tests conducted for the RMA bulk mustard disposal program. The compound  $\text{NO}_x$  was present, but not in quantities that would indicate a serious pollution problem.



The next step was to move to a full-scale incineration test at RMA, the proposed waste disposal site. Because only the limited quantity of waste generated during the pilot program was available, instead of burning at 2 gal/min, the test was restricted to burning at 1 gal/min. The waste from runs GID No. 2, GID No. 4, and GID No. 18 were used. A drum of MEA was used during furnace start-up. The results of this test showed that the waste could be burned easily and cleanly in the RMA incinerator complex. The compounds  $\text{NO}_x$ ,  $\text{SO}_2$ , and mustard and opacity were monitored in the stack, and mustard and MEA were monitored in the work area. No emissions exceeded the air quality standards, and there appears to be every indication that increasing the burning rate to 2 gal/min would also be successful. The compound  $\text{NO}_x$  is the limiting pollutant. Low levels of  $\text{SO}_2$  were detected; however, no mustard or MEA could be found. Detailed results of both the small-scale and large-scale incineration tests are provided in appendix F.

C. Monoethanolamine (MEA) and Supertropical Bleach (STB) Compatibility.

The compatibility of MEA and STB was run to determine how these two chemical decontaminants interact because of their possible simultaneous use during the demilitarization project. The compound STB was to be used outside of the cabinet in the event of any spill. The test determined that a dilute solution (5%) of STB in water when mixed with MEA created only a minor temperature rise of 4° to 12°C. When a 50% slurry of STB in water was added to an equal volume of MEA, the slurry turned dark in color and rose in temperature from 25° to 115°C. Finally, when dry, powdered STB was mixed with MEA, a spontaneous fire quickly developed. As a result, the demilitarization plan specified segregating these decontaminants and using only dilute STB solutions inside the working area where MEA would be present. A test report is included in appendix G.

D. One-Ton Container Backfill Program.

This test program was planned to determine the time and temperature parameters involved in backfilling with MEA a drained but contaminated one-ton mustard container. The test was set up in August and September 1975 and was to have been completed in September of 1975. Special thermocouples had been fabricated to monitor the temperature inside the one-ton container. The outside temperature and interior pressure were to be monitored as well.

Physically, the one-ton container was located identically to the way it was to have been located in the demilitarization unit. One valve was to be hooked to the inlet for the MEA flush, and the other valve was to be open to a vent. The one-ton container to be used had been randomly selected at Edgewood Arsenal from several one-ton containers that had been previously emptied of mustard but were still contaminated.

The procedure called for the one-ton container to be filled in two 50-gallon increments without external cooling of the one-ton container. The second increment was not to be fed until the container had cooled. The time required was to be monitored and samples of the waste were to be taken at intervals after the last increment had cooled, to check for any residual mustard.

Due to the 10 September 1975 cutoff date on project funds, no further work could be carried out. The experiment was consequently disassembled and the one-ton container was returned to the chemical agent storage yard. No data were collected.

E. Debug Program.

This program was planned to extend from January 1976 to May 1976. It envisioned three actual phases: a simulated test of the completed demilitarization equipment at Edgewood Arsenal, a simulated test of the equipment after emplacement on-site at Fort McClellan, and an agent test of the equipment at Fort McClellan by demilitarizing one one-ton container. Again, because of the project delay and subsequent cancellation, the program never reached this stage.

VII. PROJECT PERSONNEL STRUCTURE AND FIELD SETUP.

A. Project Personnel Structure.

The projected structure and quantity of personnel needed to operate the demilitarization site were to be kept to a minimum. Figure A-14 shows the proposed administrative structure.

The number of personnel that were to be trained and the number of personnel that were to be present during normal operations are noted in table 2.

Table 2. Proposed Numbers of Personnel

Title	Number trained	Number for operations	Supplying organization
Project administrator	1	1	Fort McClellan
Operations supervisor	1	1	Anniston Army Depot
Laboratory supervisor	1	1	Anniston Army Depot
Operations foreman	1	1	Anniston Army Depot
Decontamination support OIC	1	1	548th S & S Battalion
Laboratory team	3	2	Anniston Army Depot
Demilitarization operators	3	2	Anniston Army Depot
Detection team	3	2	Anniston Army Depot
Decontamination support NCOIC	1	1	548th S & S Battalion
Hot line team	10	5	548th S & S Battalion
Decontamination truck team	10	5	548th S & S Battalion
Medical team	4	2	Fort McClellan
Guards	<u>6</u>	<u>2</u>	Fort McClellan
Total	45	26	

The personnel required were to include those needed for demilitarization operations and those needed for emergency. The personnel actually operating the site during the normal working day were to be the operations supervisor, laboratory supervisor, laboratory team, operations foreman, demilitarization operators, detection team, and guard team. The remaining personnel were to be stationed at the operations control center (building T-796) outside the hazard zone area. These people were to consist of the project administrator, decontamination support OIC and NCOIC, one hot line team, one decontamination truck team, and one medical team. These people were to handle the day-to-day administrative duties of the project and were to respond to any emergencies.

A second decontamination truck team and a second hot line team were to be trained and were to be available from other duties on post in the event of an extreme emergency, and a backup medical team was to be available on call. In addition, two doctors at Noble Army Hospital, Fort McClellan, were trained in toxic agent care specifically for this project.

Extra guards were to be required to train so that two could be used during daily operations and two could be used for each of the remaining eight-hour shifts. Twenty-four-hour guarding of the yard was to be maintained until all mustard agent was detoxified.

Lastly, engineering, facilities, and safety support were to be provided as needed by personnel from Fort McClellan, Anniston Army Depot, and Edgewood Arsenal.

#### B. Field Setup.

The demilitarization site (see figure A-15) was located in the old toxic storage yard. A hazard zone analysis<sup>2</sup> for this operation and site was published in September 1974 and was subsequently approved.

The old toxic storage yard was divided into five fenced areas as indicated in figure A-15. Each of these areas was to be used with the exception of area 5, which was to be closed off.

Area 1 was to be the chemical laboratory and waste storage area. Building E4456 (figure A-16) was to be used as the chemical laboratory and was to house items such as a fume hood, a gas-liquid chromatograph, and all chemicals necessary to do analysis of the detoxified waste and bubblers providing the air-monitoring capability. A gravel pad was to be constructed in the vicinity of building E4454 for holding the waste liquid in 55-gallon drums and for holding the one-ton containers pending shipment to RMA.

Area 2 was to be the main demilitarization area. The demilitarization equipment was to sit on the concrete pad (figure A-17) in the center of this area. A Butler-type building without sides was to extend over the majority of the pad to shelter the equipment and operational personnel. This was called the pavilion structure. Next to the pavilion structure was to be the thaw tent where the mustard-filled one-ton containers were to be stored. Also in this area were

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<sup>2</sup>Solomon, Irving. Demilitarization of One-Ton Containers of Mustard Agent (HD) at Fort McClellan, Alabama; Hazard Zone Analysis. September 1974.



to be gravel pads for the filter solids bath and the MEA 55-gallon-drum storage area. Other facilities such as a work bench, two hot lines with exits from the enclosed area, and emergency deluge showers and eyewash fountains were also to be provided to the workers in this area.

Area 3 was to be the personnel support area. Building E4457, the only existing building there, housed a water heater. Near this building were to be placed two general purpose (GP) medium tents. One tent was to be used as the change tent, the second was to be equipped as a shower tent and hooked into the water heater in building E4457. A third GP medium tent was to be placed close to the exit from area 2 as part of the hot line and was to serve as the clothing drop tent. A lime pit for shower water runoff was also to be provided in this area. Laundry facilities were to be provided by Anniston Army Depot on a daily pickup basis.

Area 4 was to be the immediate support area for all the other areas. The outside gate in this area was to be the main entrance to the yard and was to have a guardpost. Building E4450 (figure A-18) was to serve as an administrative building for the yard area with a desk for the operations supervisor and with facilities for the workers for eating and smoking. Building E4450 also had lavatory fixtures and an existing septic system. Building E4453 was to serve as the STB storage and mixing area. This was to keep STB separated from any possible contact with MEA, except in dilute solution form (see section VI.C).

Thus, area 2 was to serve as the main demilitarization and chemical exclusion area. The remainder of the yard was to support the main operations. The remaining areas would have been operated as a chemical limited area.

## VIII. EQUIPMENT LAYAWAY AND DISPOSITIONS.

### A. Background.

On 10 September 1975 the Department of the Army directed that the demilitarization effort on the disposal of one-ton containers of mustard agent (HD) at Fort McClellan, Alabama, be held in abeyance. This was to make a decision on the alternatives of disposing of the agent onsite or of moving the agent to an alternate storage or disposal site. In October 1975, the decision was made to pursue the movement or collocation alternative. Consequently, it was directed that the unfinished equipment be laid into storage or committed to other programs at the discretion of the AMC Program Manager for Demilitarization of Chemical Materiel.

### B. Layaway.

The unfinished demilitarization equipment was placed in storage as directed along with other components that could not immediately be used on other programs. The major equipment that remains stored is as follows:

1. One-ton container cabinet with load cells, weight platform, and trolley unit
2. Reactor cabinet, unfinished
3. Reactor, 500-gallon stainless steel, with four internal baffles, jacketed with seven external baffles

4. Reactor piping, stainless steel, with appropriate ball valves, also stainless steel, Teflon packed
5. Transfer tank, stainless steel, approximately 30 gallons
6. Surge tank, stainless steel, removable head, approximately 55 gallons
7. Pump, 10 gal/min MEA-transfer, Viking model GG 195D; with direct drive, 1800 rpm, 1/2-hp motor, explosion-proof, class 1, group D
8. Pump, 20 gal/min, waste-transfer, Viking model KK 624; with 1150 rpm, 3/4-hp motor, explosion-proof, class 1, group D
9. Vacuum pumps, 3 each, General Rand Reciprotor model 506R
10. Fan, explosion-proof
11. Nitrogen purge equipment
12. Agitator, Lightning model N33 G-75, 3/4 hp, explosion-proof, class 1, group D; with 61-inch stainless steel shaft and mixing propellers
13. Heat monitoring equipment, Honeywell, with strip charts, recorders, and thermocouples.

C. Disposition.

Some major items that were procured for the Fort McClellan operation were given to other laboratory or demilitarization programs. Still other ordered items were cancelled, such as the major laboratory equipment that was to be installed at Fort McClellan. Items that were transferred to other programs are as follows:

<u>Item</u>	<u>Program</u>
Drums, 55-gallon, MEA-filled, 50 each	ID set disposal
Drums, 55-gallon, waste storage, empty, 200 each	ID set disposal
Gas-liquid chromatograph with associated equipment	Pine Bluff Arsenal Laboratories
Analytical chemicals, various	ID set disposal
Gloves, butyl rubber	ID set disposal
Filter units, M6A2, with Teflon lines hose	Omnibus disposal

The future disposition of any remaining equipment remains the responsibility of the DA Program Manager for Chemical Demilitarization and Installation Restoration (the former AMC Program Manager for Demilitarization of Chemical Materiel).



## IX. COST AND SCHEDULE SUMMARY.

### A. Cost.

The cost estimate for the demilitarization and disposal operations at Fort McClellan, Alabama, was being updated when the project was held in abeyance. The previous estimate for project completion, reported on 6 May 1975, was \$847,300 including a 15% reserve. This breakout was made in table 3.

Table 3. Cost Estimate Summary (Thousands)

Task	FY74	FY75	FY76	FY7T	FY77
Planning studies	11	53.6	0.5	-	-
Facilities	-	124.5	17.5	-	-
Project support	-	45.5	182.0	23.0	10.0
Operations	-	16.0	86.0	49.7	-
Cleanup	-	-	-	38.0	85.0
Final report	-	-	-	16.7	10.3
Total	11	239.6	286.0	127.4	105.3

Actions were directed after 6 May 1975 to include the cost of demilitarizing 43 mustard-filled 155mm rounds and the use of the TRACOR real-time alarm in the operations. These factors were never included in the final costs.

The cost of the project through to completion of the layaway, disposition, and writing of the final report (see table 4) is \$345,600.

### B. Schedule.

The Fort McClellan mustard disposal program was managed with a PERT-style diagram that showed major events on individual lines laid out over a grid which, with dates, could be used to determine if each event was on schedule. The major events listed were as follows:

1. Basic plan staffing
2. Environmental impact assessment staffing
3. Public information package staffing

Table 4. Actual Cost (Thousands)

Task	FY74	FY75	FY76	FY7T
Planning studies	11	53.6	6.5	—
Facilities	—	124.5	30.5	—
Project support	—	45.5	48.0	—
Operations	—	16.0	—	—
Cleanup	—	—	—	—
Final report	—	—	—	<u>10.0</u>
Subtotal	11	239.6	85.0	10.0

4. Hazard zone calculations staffing
5. Fault tree hazard analysis staffing
6. Standard operating procedures development
7. Blueprint drawing documentation
8. Procurement for fabrication
9. Fabrication of demilitarization equipment
10. Procurement for equipment related items
11. Procurement for site and laboratory items
12. Procurement of monoethanolamine
13. Training program and coordination
14. Site preparation
15. Demilitarization operations
16. Waste incineration
17. Final report and film.

The planning stage for these events began in January 1974 and was fully implemented by July 1974. The project was to reach the point where tentative approval for an agent test using a single one-ton container was to be granted by April 1976. The test was to last through May 1976 and changes made to any procedures were to be made by, and operations commenced in, July 1976. Operations, including site clean-up, were to be completed by October 1976. The incineration of the waste liquid was to be completed by November 1976. The final documentation of the project was to be completed by January 1977.

When the project closed in September 1975, all elements of the PERT were on or ahead of schedule. The approval of essential documentation such as the Basic Plan<sup>3</sup> and the Environmental Impact Assessment<sup>4</sup> was one month ahead of schedule when the project was stopped by the Department of the Army. The staffing and approval of these documents was the project critical path. Since that date, all project equipment has been laid away or transferred to other programs as described in section VIII. The last project action scheduled and funded is the completion of this report.

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<sup>3</sup>Brankowitz, W. R. Basic Plan for the Demilitarization of One-Ton Containers of Mustard Agent (HD) at Fort McClellan, Alabama. Office of the AMC Program Manager for Demilitarization of Chemical Materiel. 15 August 1975.

<sup>4</sup>Brankowitz, W. R. Environmental Impact Assessment for the Demilitarization of One-Ton Containers of Mustard Agent (HD) at Fort McClellan, Alabama. Office of the AMC Program Manager for Demilitarization of Chemical Materiel. 15 August 1975.



APPENDIX A

FIGURES



Figure A-1. One-Ton Containers at Fort McClellan

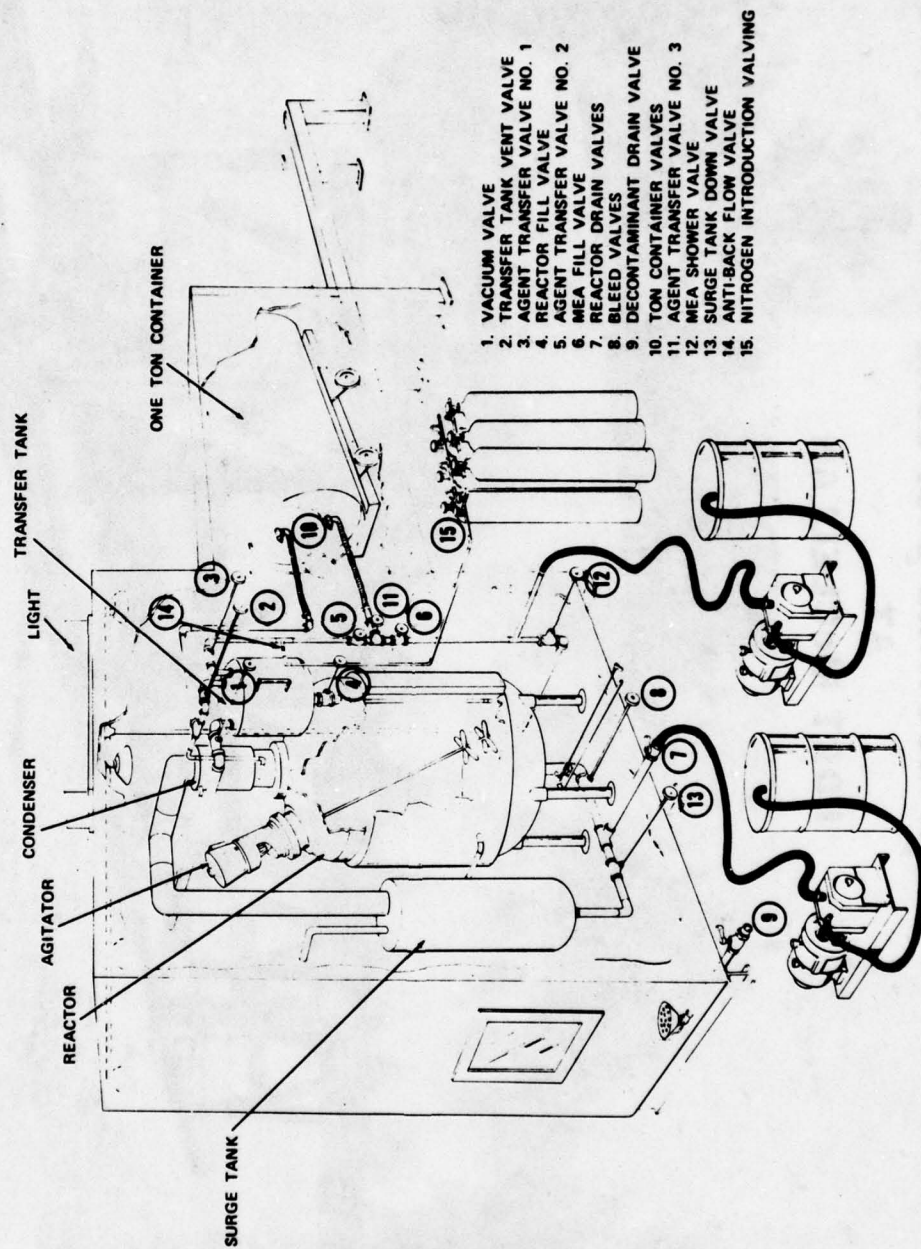


Figure A-2. Mustard Demilitarization Equipment - Front View

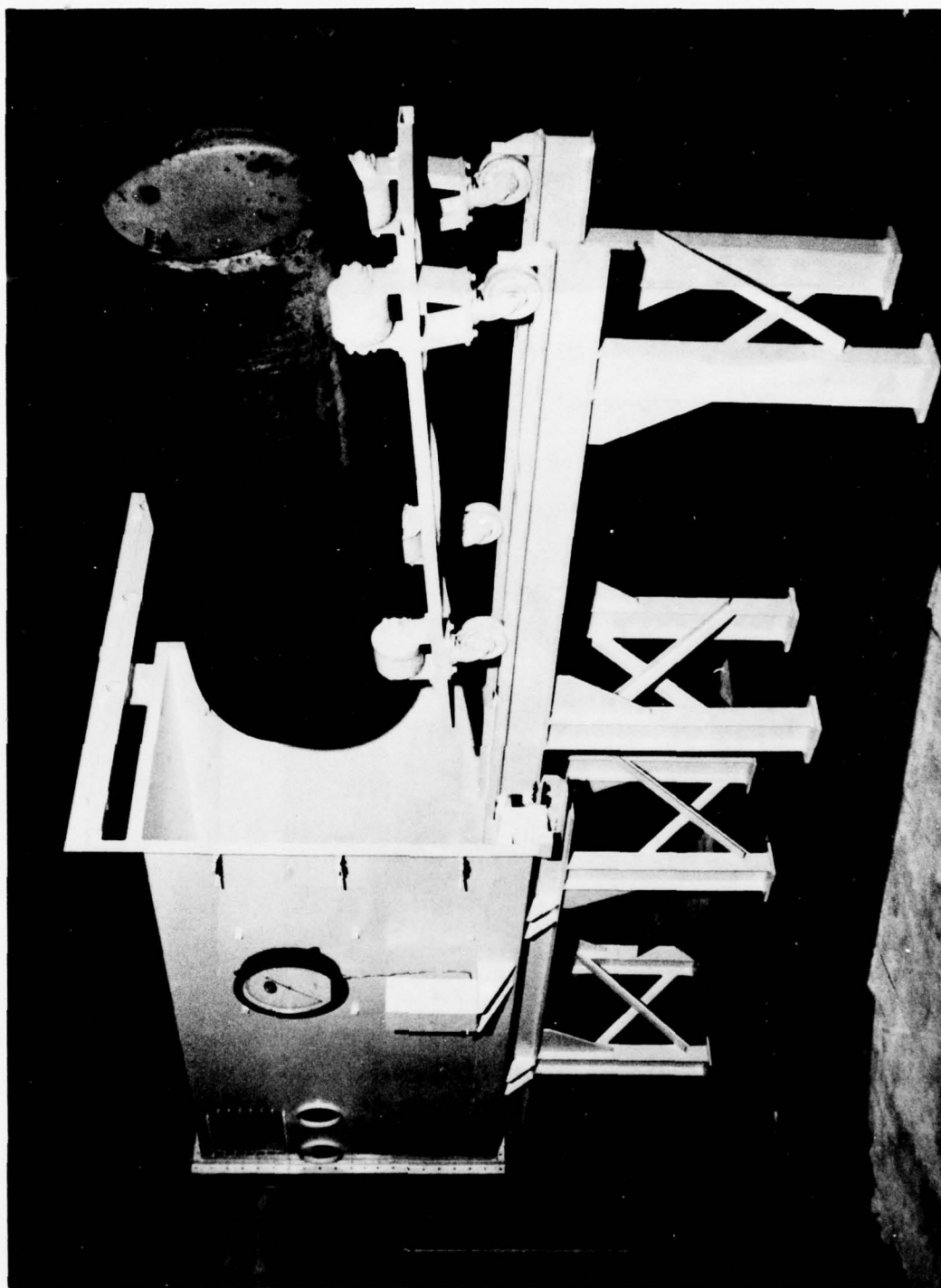


Figure A-3. One-Ton Container Cabinet with One-Ton Container in Place

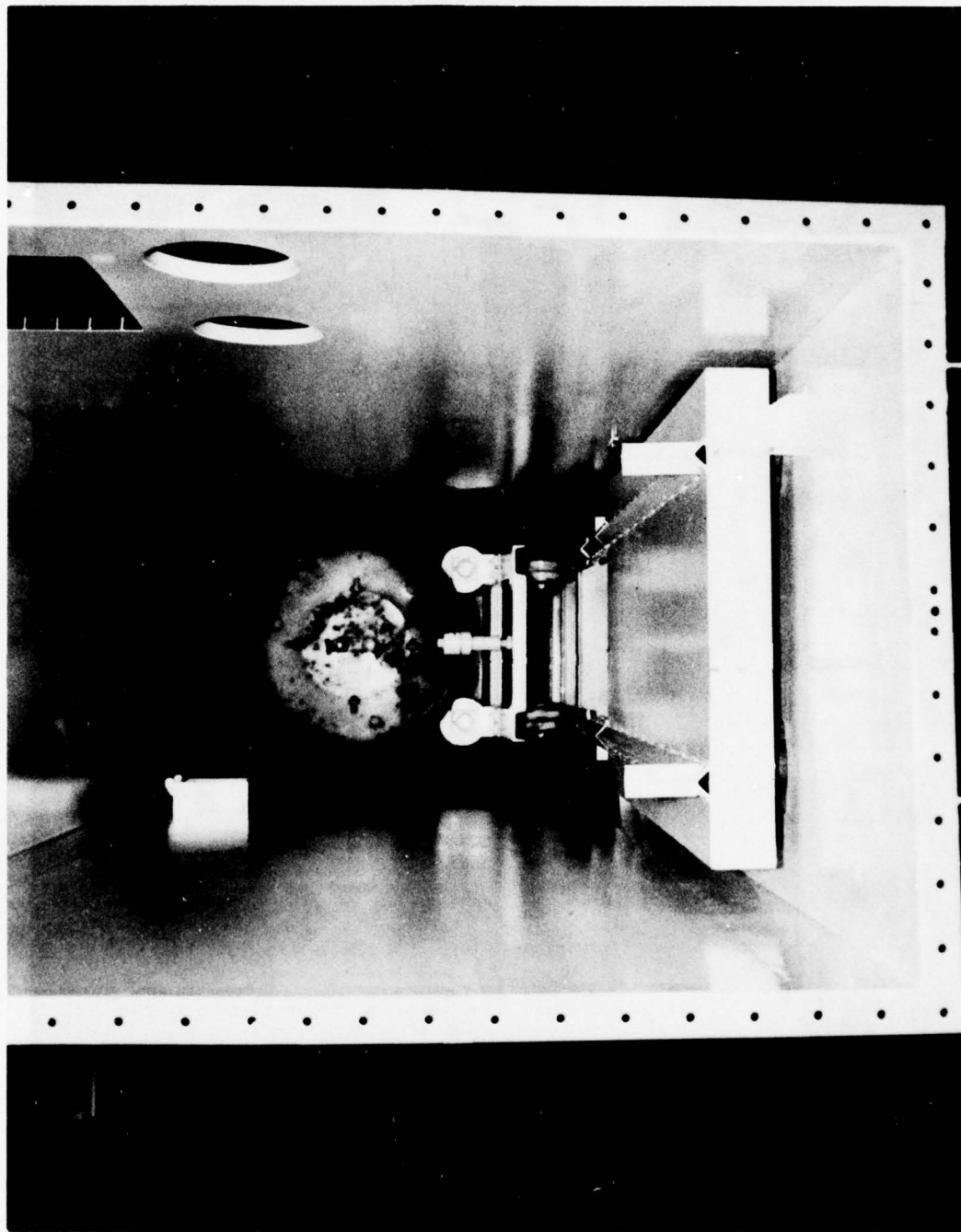


Figure A-4. Weigh Platform in One-Ton Container Cabinet



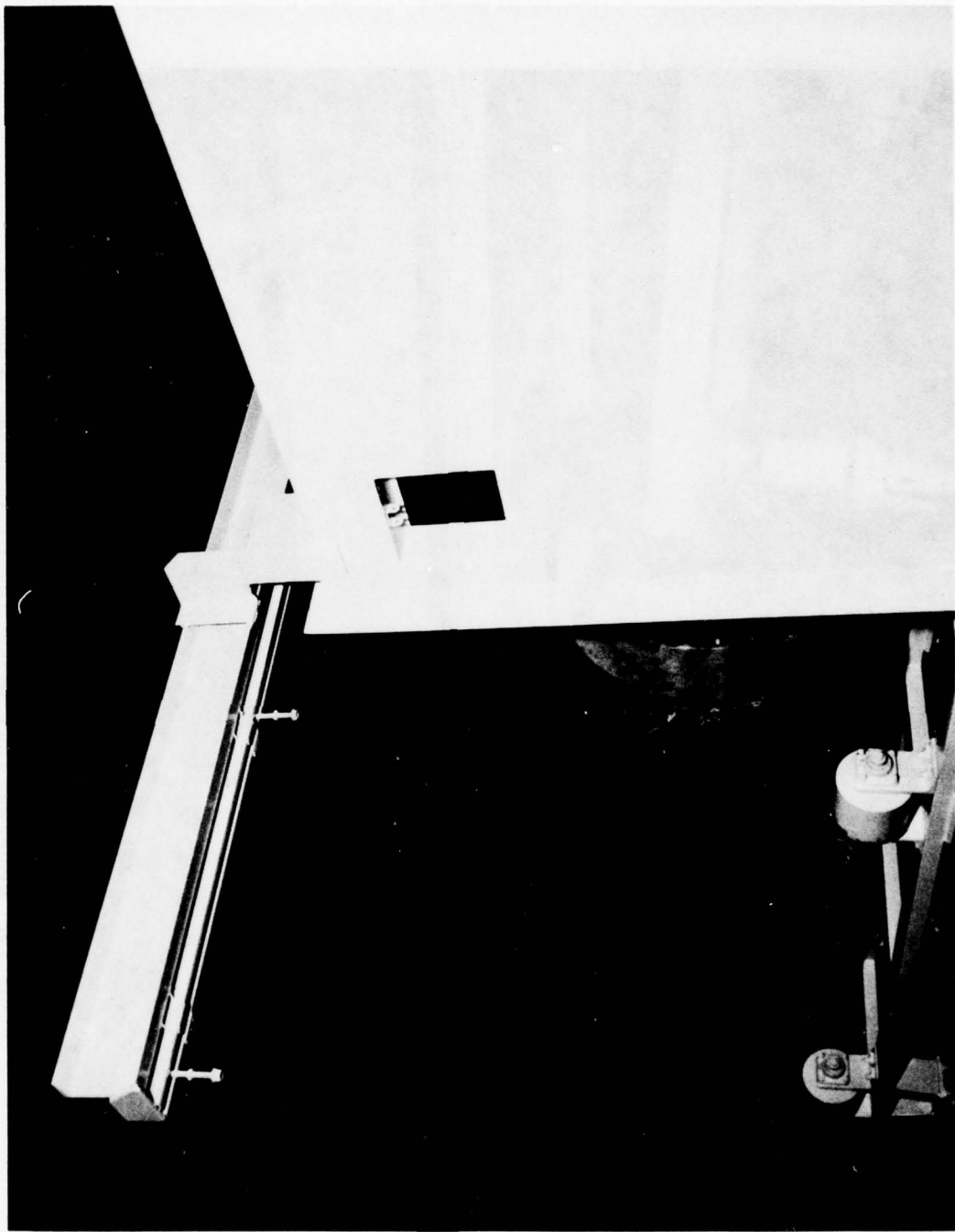


Figure A-5. Door Assembly (Door Removed for Storage) of One-Ton Container Cabinet



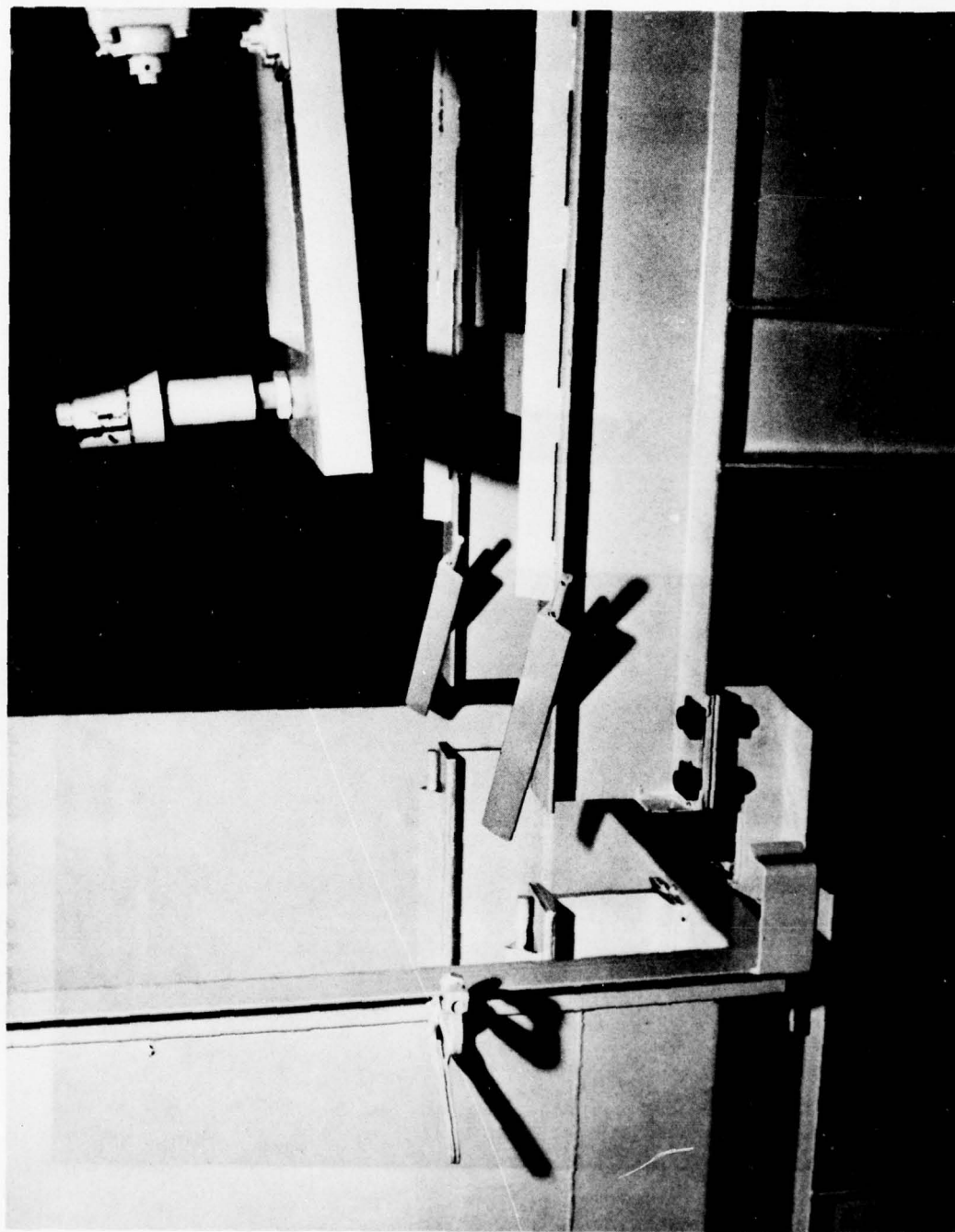


Figure A-6. Removable Track Section, One-Ton Container Cabinet

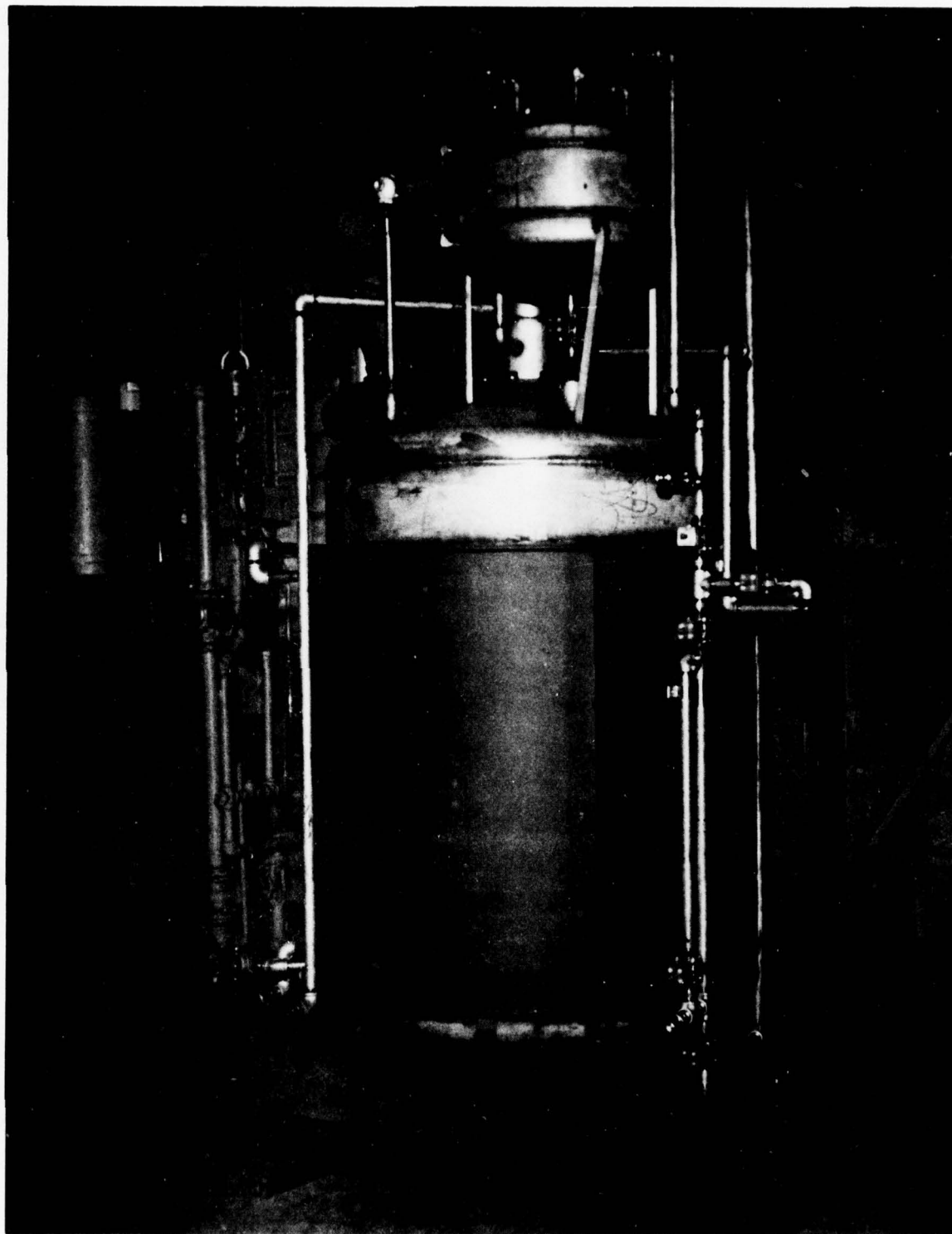


Figure A-7. Reactor with Transfer Tank and Process Piping – Front View

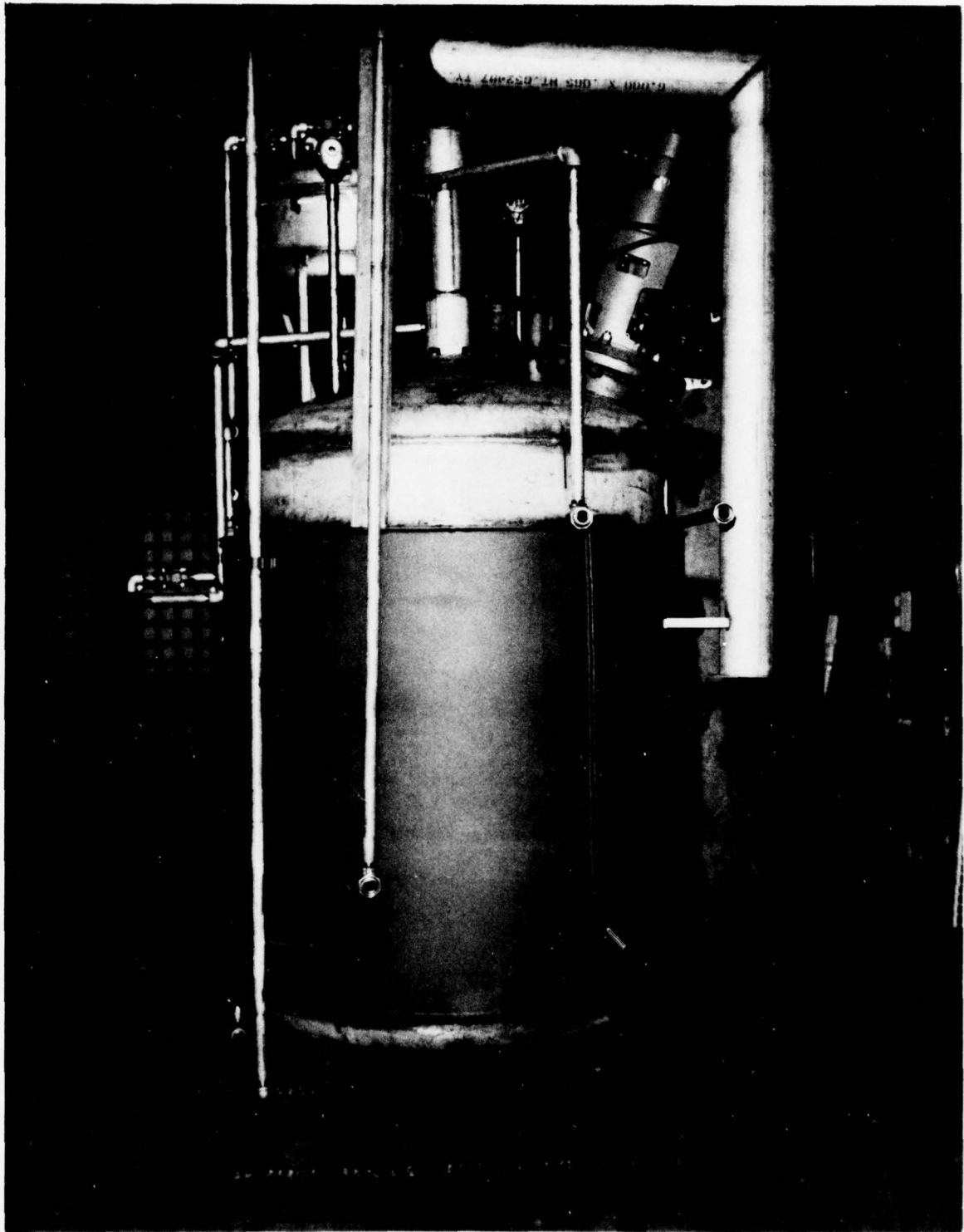


Figure A-8. Reactor with Transfer Tank and Process Piping – Rear View



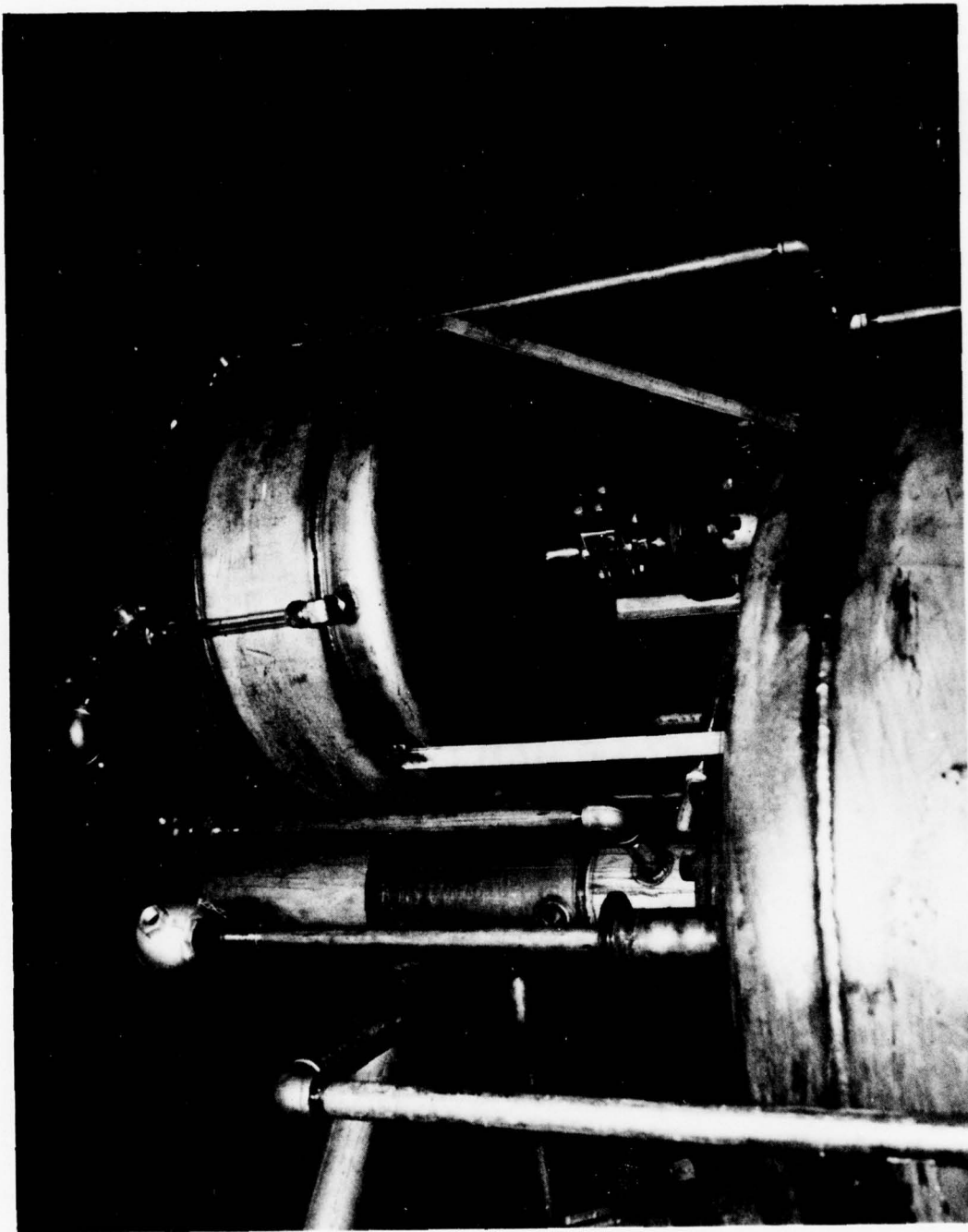


Figure A-9. Transfer Tank, Condenser, and Front Thermocouple

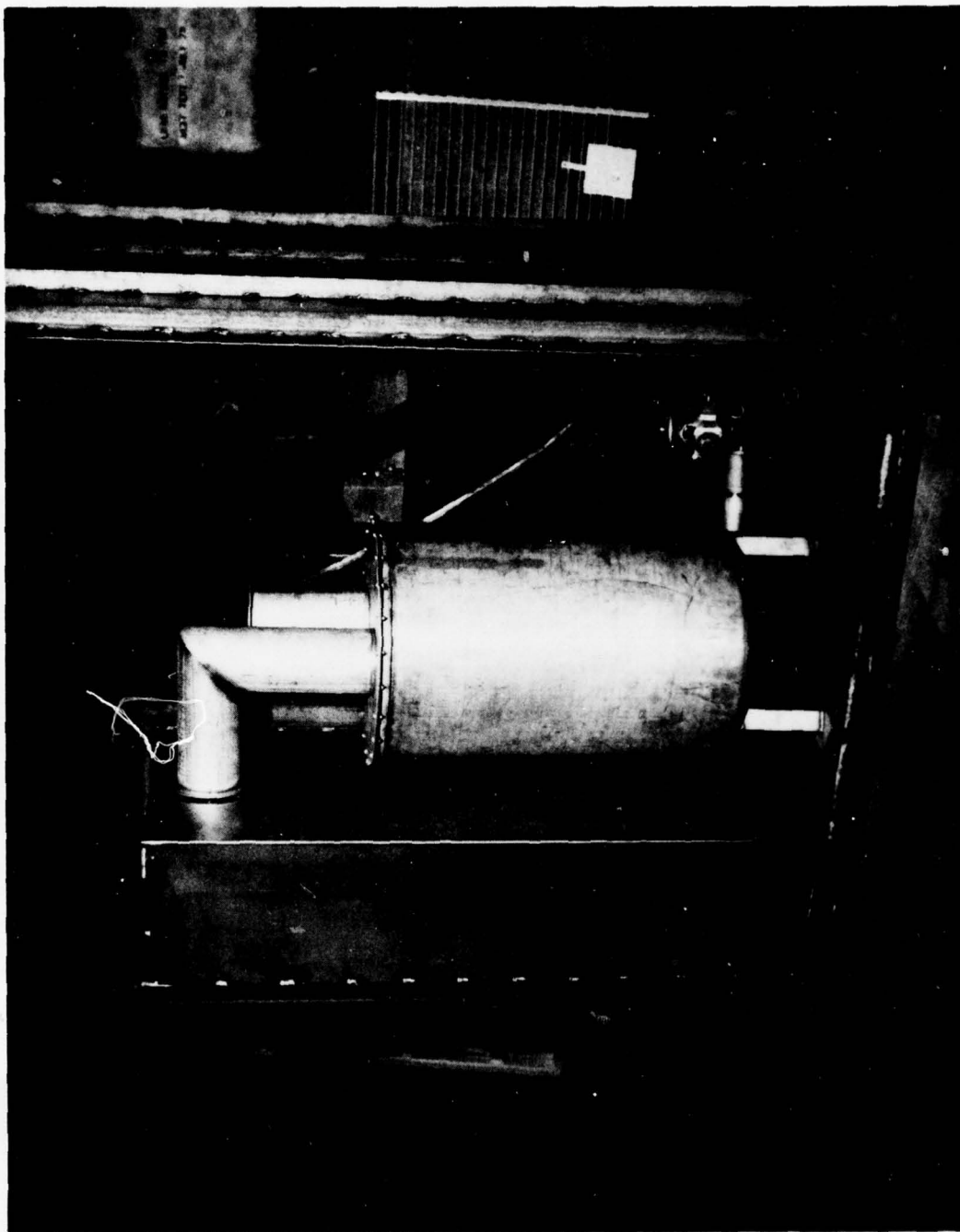


Figure A-10. Reactor Cabinet with Surge Tank, Cabinet Top, and Other Incomplete Parts Inside

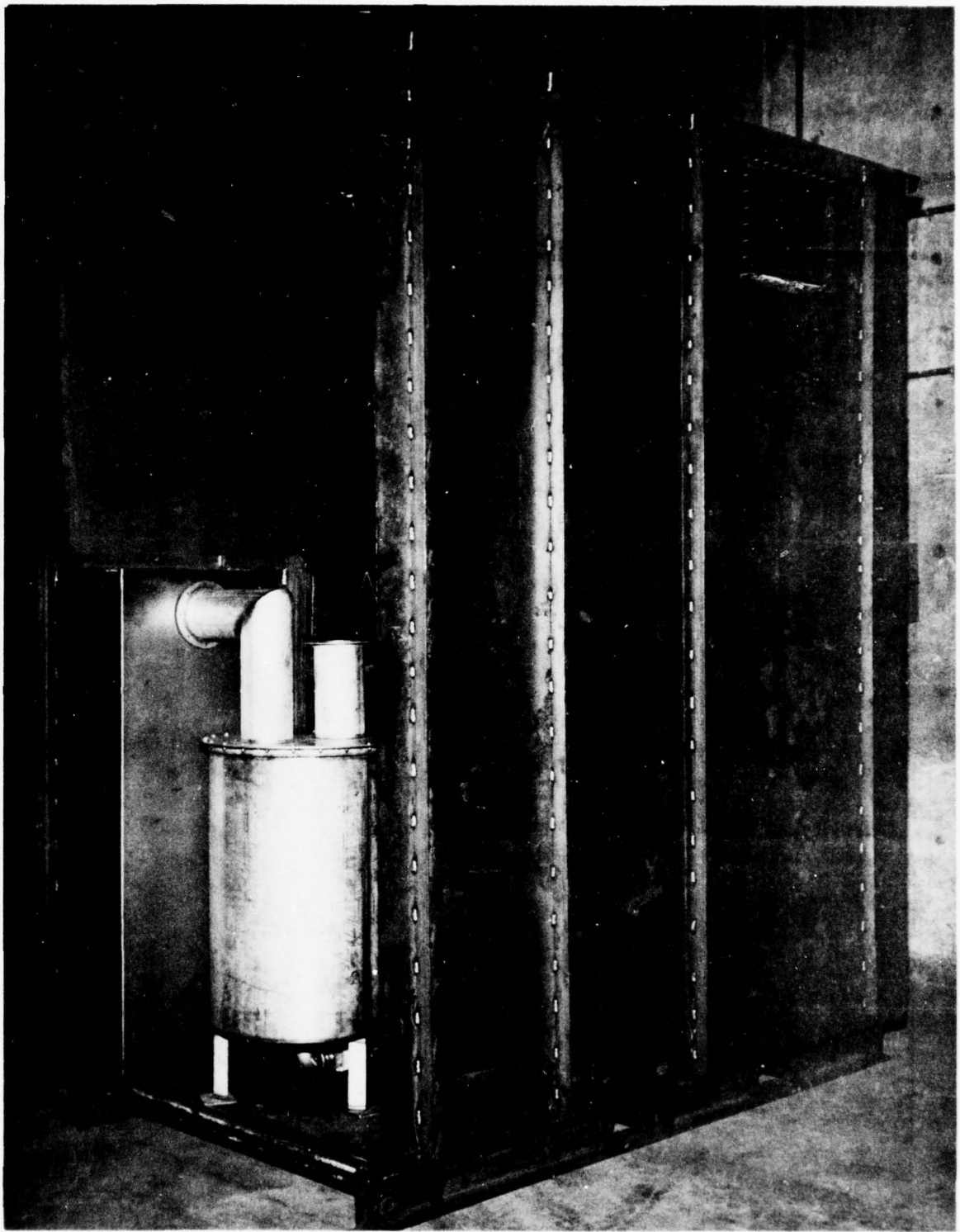


Figure A-11. Reactor Cabinet — Front View



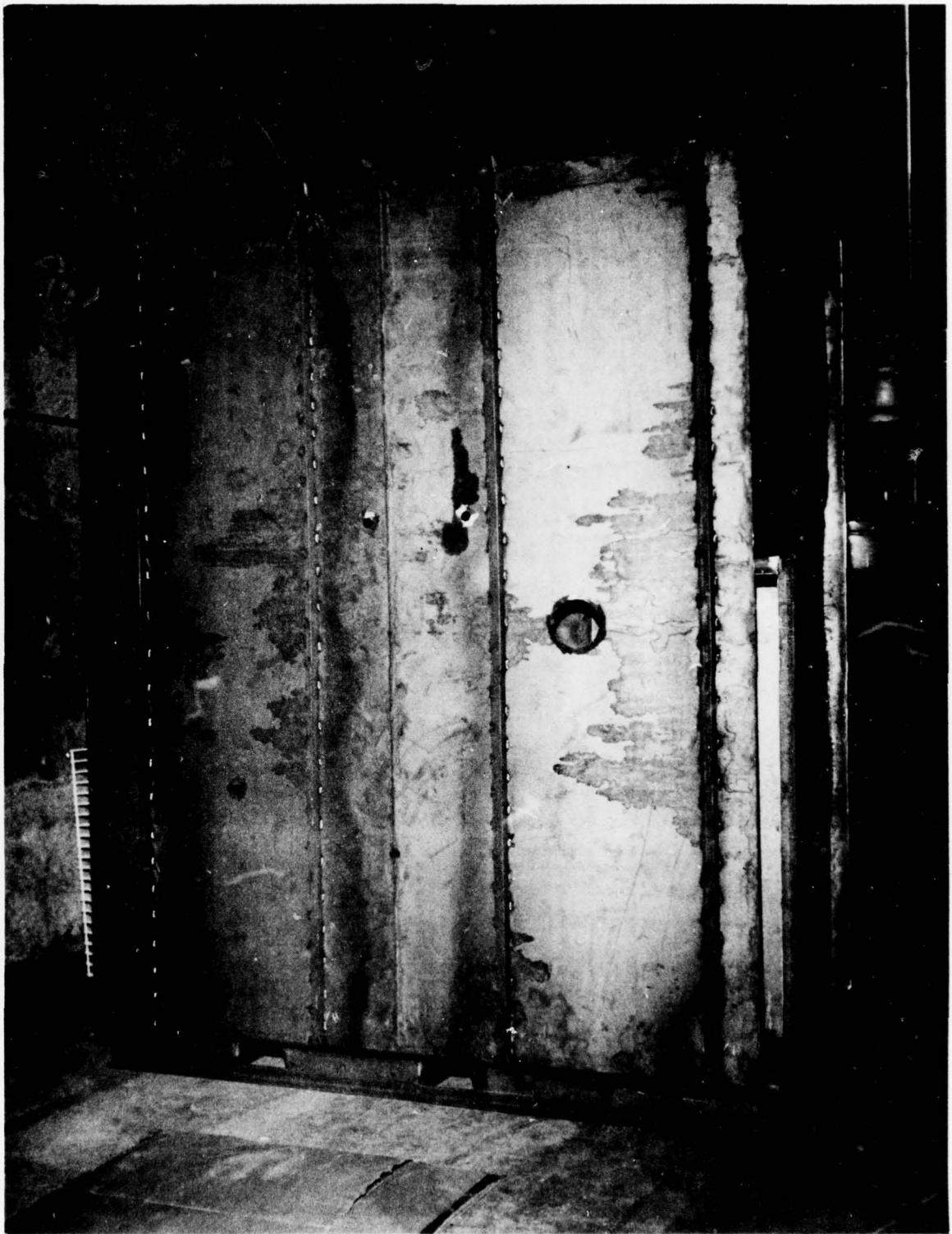


Figure A-12. Reactor Cabinet – Rear View

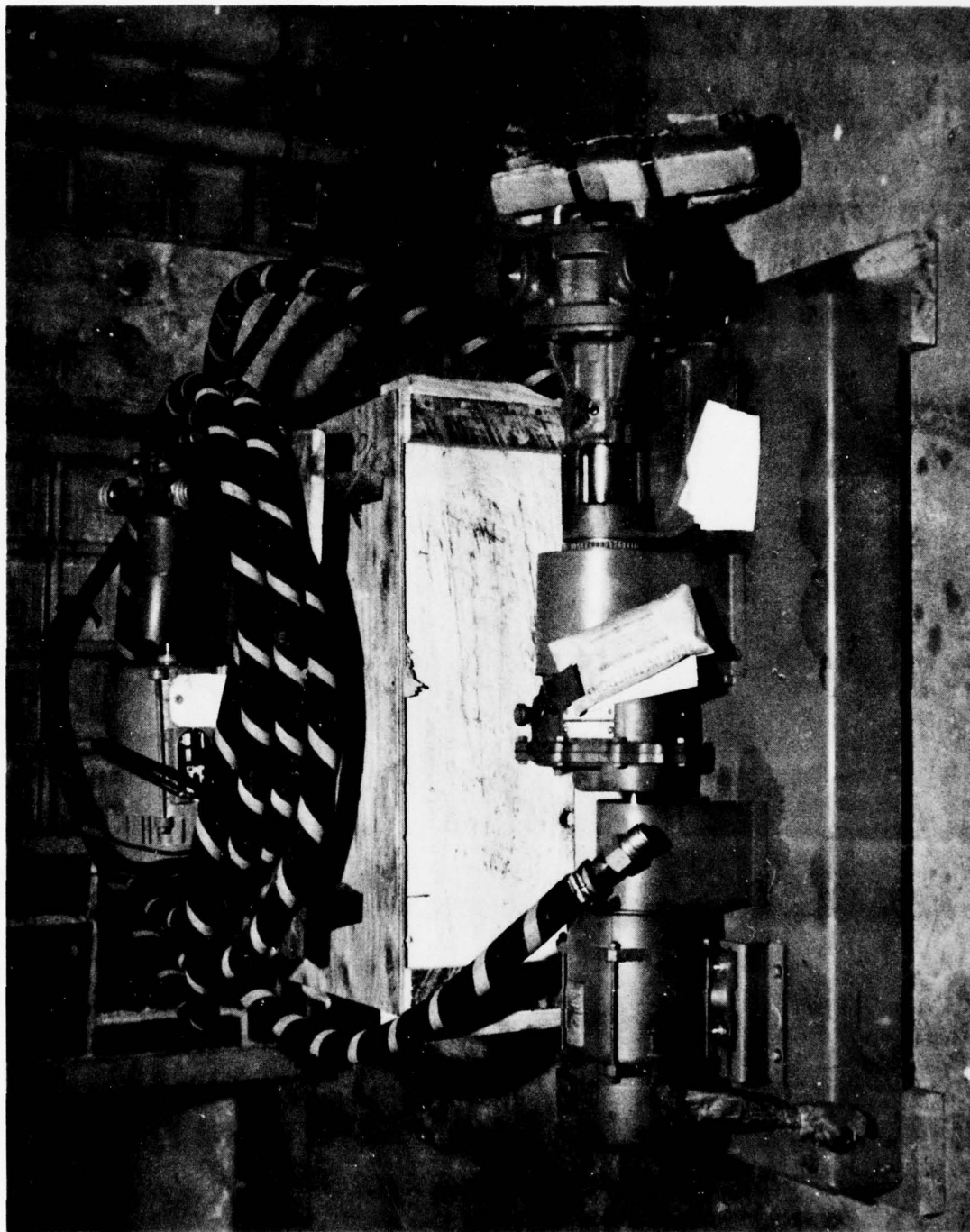


Figure A-13. Process Feed Pump (Top) and Waste Drain Pump (Below) with Attendant Chemical Hose

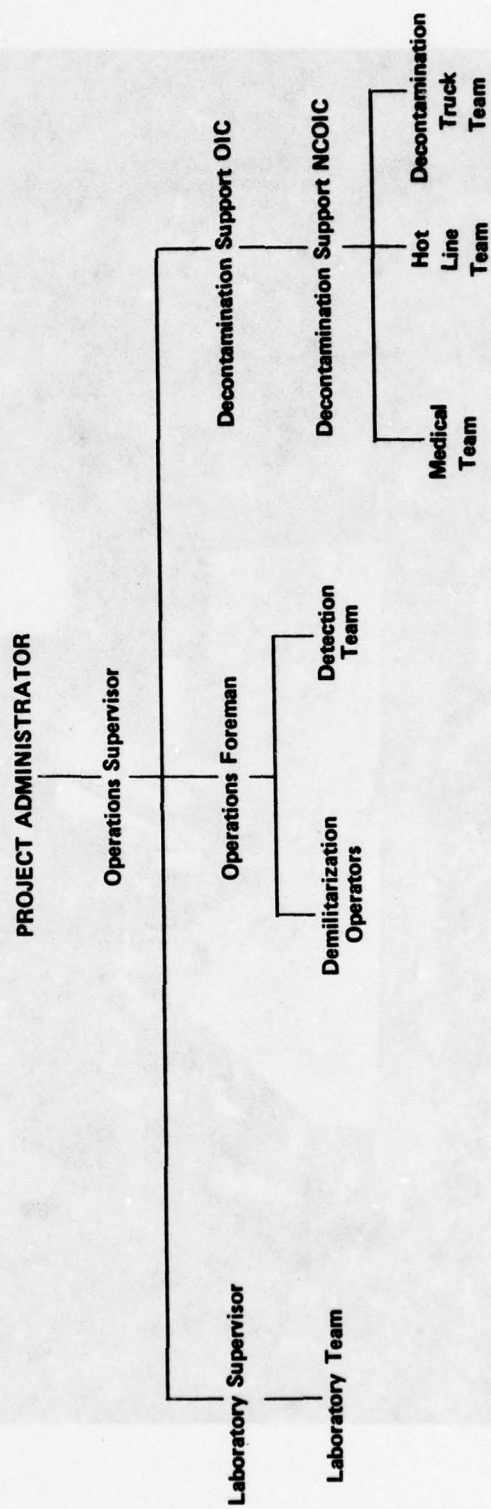


Figure A-14. Proposed Personnel Structure



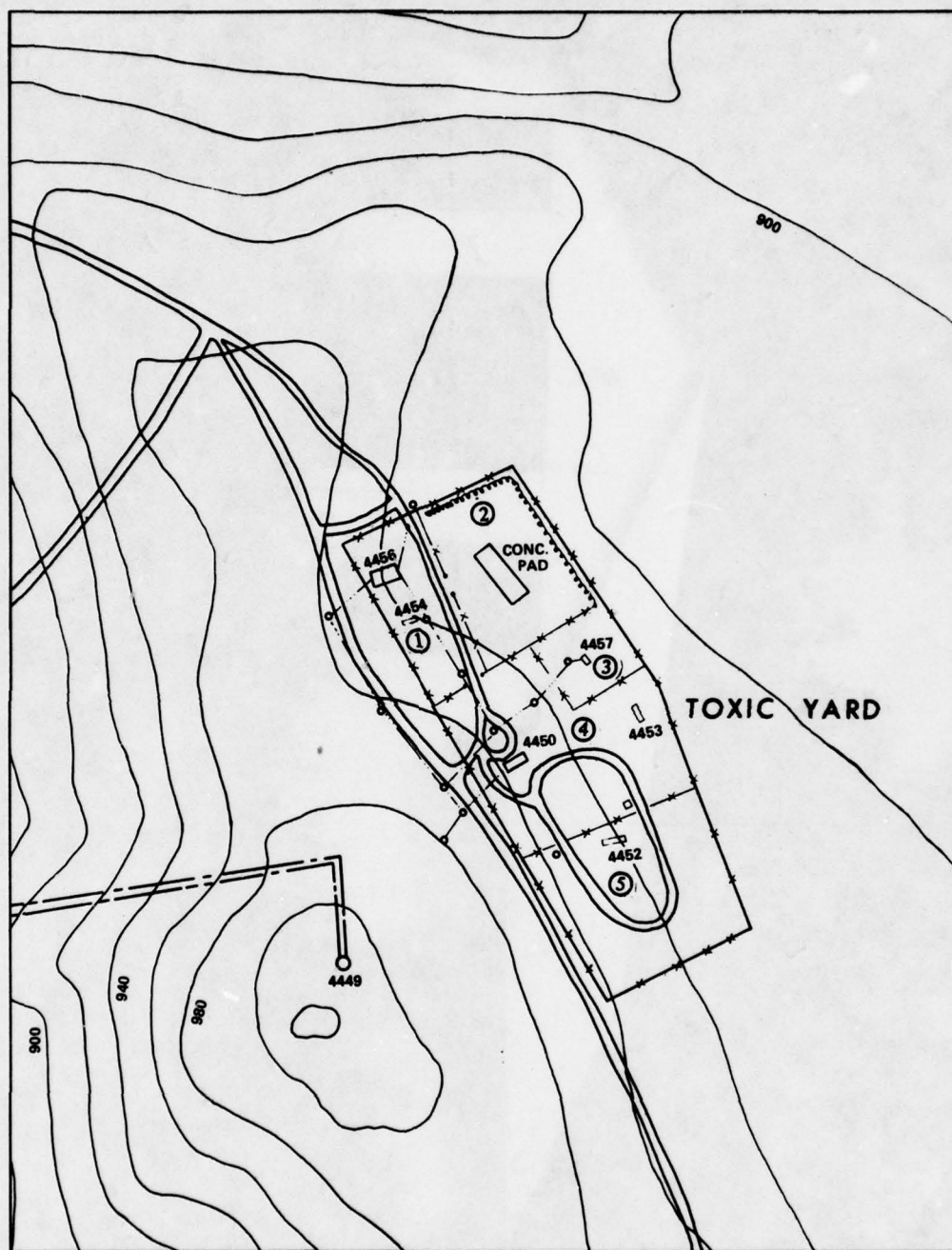


Figure A-15. Demilitarization Site Map



Figure A-16. Proposed Laboratory Building

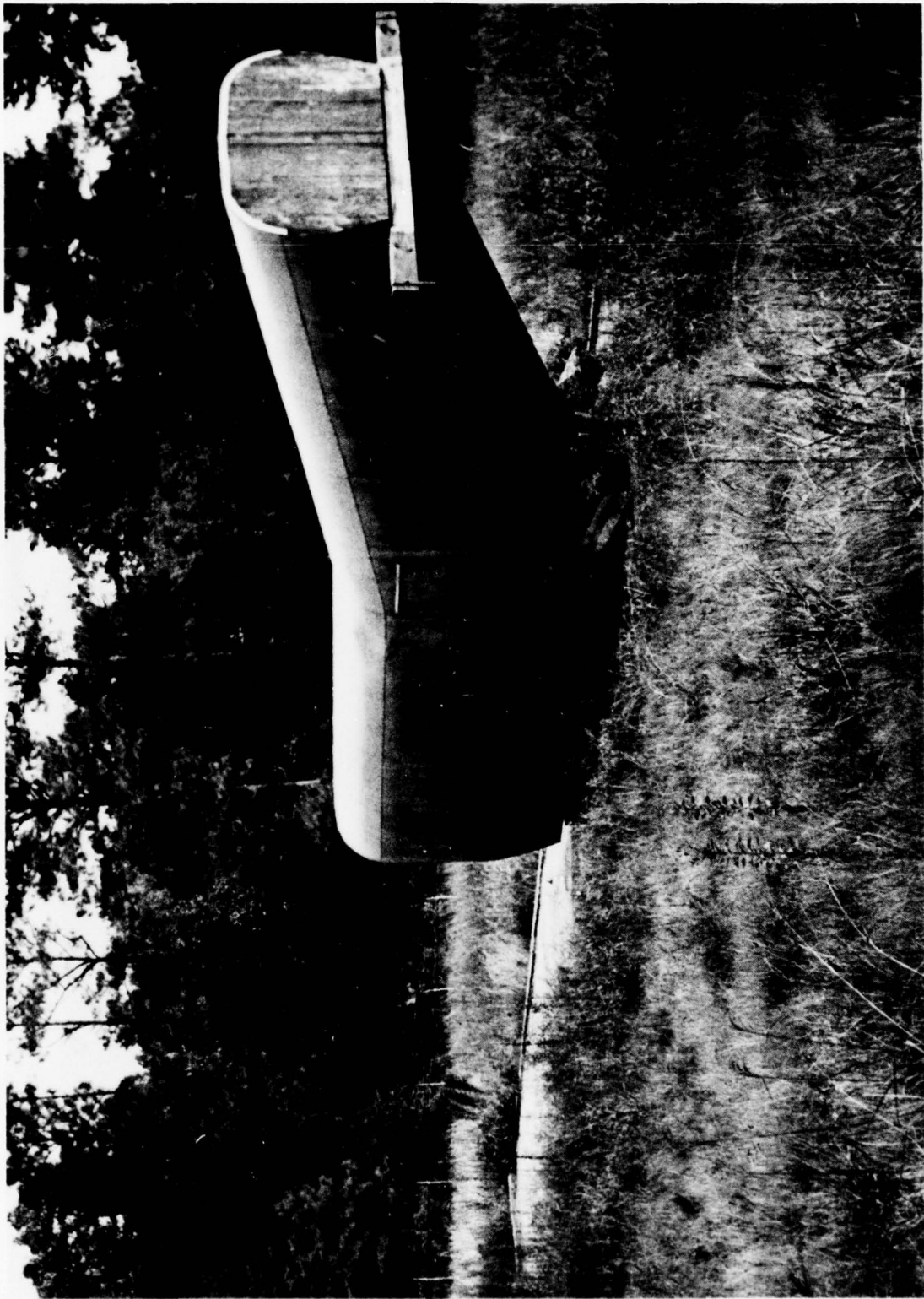


Figure A-17. Concrete Pad Area





Figure A-18. Proposed Site Administrative Area

## APPENDIX B

### LABORATORY REPORT ON MONOETHANOLAMINE

Initial tests involving monoethanolamine (MEA) and mustard were conducted by the Chemical Laboratory at Edgewood Arsenal. These tests were made in comparison with other accepted decontaminants. The initial tests were performed for another demilitarization program (Demilitarization of Obsolete Toxic Gas Sets); however, they were directly applicable to the Fort McClellan program as the agent addressed was mustard, type HD. A summary of these initial tests is included herein.

#### I. INTRODUCTION.

A study was initiated in an effort to find a suitable decontaminant for neat 2,2'-dichlorodiethylsulfide (H) and its distilled form (HD) contained in the K941 and K942 toxic gas sets. This work was conducted as part of a larger study to find suitable decontaminants for various agents contained either neat in solution, or impregnated in carbon and plastic surfaces in obsolete chemical agent identification training kits. After discussions with various personnel, it was concluded that the requirements for optimum decontamination conditions should be as follows:

1. The decontaminant should be
  - a. Nontoxic
  - b. Noncorrosive
  - c. Inexpensive
  - d. Of high boiling point
  - e. Of high flash point
  - f. Nonflammable.
2. The decontamination reaction should have the following requisites:
  - a. The rate of the reaction should be relatively fast.
  - b. Minimal quantities of reactants and solvents should be required.
  - c. The reaction conditions should be homogeneous (one-phase system).
  - d. Relatively simple equipment should allow the reaction to be performed under field conditions.
  - e. Nonprofessional personnel should be able to conduct the detoxification process.

f. The reaction products formed should conform to a Department of Transportation (DOT) toxicity test classification of class B poison or less.

g. Ease of disposal of the decontamination products.

Several known decontaminants for H were considered as not meeting the above criteria. The conventional military decontaminant DS-2 (a mixture of 2% sodium hydroxide, 28% methylcellosolve, and 70% diethylenetriamine) was not considered a suitable decontaminant for H/HD for the following reasons:

1. A large amount of decontaminant would be required — at least 50 parts of DS-2 to one part H.
2. A potential fire hazard would be involved in this reaction.<sup>1</sup>
3. The reaction would not be homogeneous.
4. The products would leave an ash if disposed of by conventional incineration.

The use of sodium hydroxide (NaOH) as a decontaminant for H/HD was rejected for the following reasons:

The overall reaction rate of H and NaOH is too low. H/HD is only very slightly soluble in aqueous caustic solutions and the rate determining step in this heterogeneous (two-phase) system is the rate of solution of the sulfur mustard in aqueous NaOH.

2. The heterogeneity of this reaction requires very efficient mixing. The equipment to effect good mixing is complex and presents a potential problem in the design for field use.

3. Even alcoholic caustic, whose rate of reaction with H/HD is higher than that of the heterogeneous aqueous caustic reaction, is not satisfactory. The half life (11 hours) of the H/HD in alcoholic caustic is too long for field use. (Recall that seven half lives are needed to reduce the amount of H/HD to 1% of its initial value.)

4. One of the decontamination products of the H/HD—caustic reaction is the volatile divinyl sulfide. The product has been reported in the literature as being highly toxic.<sup>2,3</sup>

5. Incineration of the products of the H/HD—caustic reaction would result in the production of salt residues which would add to the burden of the disposal procedure.

6. Other possibilities, such as the addition of solvents and/or amines to the NaOH in order to afford a homogeneous reaction system, would produce a decontaminant similar to DS-2 with inherent disadvantages already cited.

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<sup>1</sup>Military Chemistry and Chemical Agents. TM 3-215. p 87. 1963.

<sup>2</sup>Helfrich and Reid. J. Am. Chem. Soc. 42, 1208 (1920).

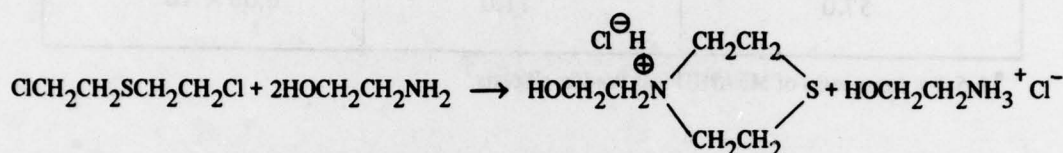
<sup>3</sup>Bales and Nickelson. J. Chem. Soc. 121, 2137 (1922).



## II. EXPERIMENTAL RESULTS AND DISCUSSION.

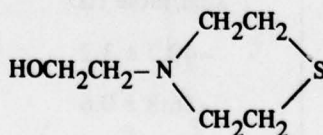
A number of potential decontaminants for HD including MEA, hydrazine, piperazine, dimethylsulfate, water/MEA/methanol, tributylamine/mercaptoethanol, tributylamine/thiophenol, sodium iodide, and potassium ethyl xanthate in a methanol water solution were tested and evaluated in accordance with the selection criteria stated previously. The majority of these compounds or mixtures were rejected for one or more reasons, such as: (1) low rate of reaction with HD; (2) formation of a secondary solid phase; (3) formation of two or more liquid phases with HD; or (4) the inherent toxicity, corrosiveness, or flammability of the decontaminant or final reaction products.

MEA appeared to meet most of the aforementioned requirements when tested with HD using a 5:1 v/v MEA/HD ratio. The general equation describing the primary reaction between MEA and HD is as follows:



On the assumption that thiomorpholine is a stronger base than MEA, this equation depicts two moles of MEA reacting with one mole of HD to produce one mole of N-(2-hydroxyethyl)thiomorpholine hydrochloride and one mole of monoethanolamine hydrochloride. A 5:1 v/v ratio of MEA/HD, the actual volume ratio of MEA/HD contemplated to be employed, actually represents approximately 10:1 mole ratio of MEA/HD. On a molar basis of 2:1 MEA/HD, the 5:1 v/v ratio provides a 400% excess of MEA. However, if in this reaction MEA is the predominant proton acceptor, then approximately three moles of MEA would be consumed. In this case, a 5:1 v/v ratio of MEA/HD would represent approximately a 230% excess of MEA. This excess of MEA is required to ensure complete reaction at a reasonable rate.

Isolation of products of the neat HD/MEA reaction was accomplished by preparative gas chromatography, distillation, and fractional precipitation techniques. Identification of the products of this reaction was done by means of vapor phase chromatography, mass spectrometry, nuclear magnetic resonance spectroscopy, and chemical microanalysis. With the exception of the monoethanolamine hydrochloride, the only major product obtained had a configuration consistent with structure I or, possibly, its hydrochloride salt.



Structure I: N-(2-hydroxyethyl)thiomorpholine

An additional minor product, bis(hydroxyethylaminoethyl)sulfide ( $\text{HOCH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2$ )<sub>2</sub>S was identified.

Kinetic studies of the MEA/H reaction were made as a function of temperature. A condensed version of these data is presented in table B-1.

Table B-1. Kinetic Data for the MEA/HD Reactions\*

Temperature	Half life ( $t_{1/2}$ )	K observed
°C	min	min <sup>-1</sup>
25.2	321.0	$2.16 \times 10^{-3}$
35.2	82.0	$8.44 \times 10^{-3}$
44.4	39.6	$1.75 \times 10^{-2}$
57.0	11.0	$6.06 \times 10^{-2}$

\* A 5:1 volume ratio of MEA/HD was used in all tests.

The results of the investigation indicate that the HD/MEA reaction proceeds fairly rapidly at moderate temperatures. The reaction is pseudo first-order with respect to H. The activation energy was calculated to be 19.8 kcal/mole HD. This value was obtained from a semilogarithmic plot of second-order rate constants (derived from reaction rate and MEA concentration data) against the reciprocal of absolute temperature over the temperature range of 25° to 57.5°C.

Heats of reaction were determined for the HD/MEA reaction at several reaction temperatures in a specially designed and constructed research calorimeter. This was accomplished by measuring the thermal rise produced by the introduction of a small amount of HD into a large amount of MEA contained in the calorimeter. These heat rise measurements (in duplicate) were made over periods of several hours to allow complete reaction of the HD with the MEA. The heats of reaction at various temperatures are shown in table B-2.

Table B-2. HD/MEA Heat of Reaction Data\*

Temperature	Average heat of reaction**	Heat capacity of products
°C	kcal/mole HD	cal/gm °C
50	$-40.7 \pm 3.2$	$0.802 \pm 0.006$
65	$-56.8 \pm 0.6$	$0.825 \pm 0.001$
77	$-60.0 \pm 1.5$	$0.816 \pm 0.004$
86	$-62.5 \pm 0.5$	$0.823 \pm 0.004$

\* HD/MEA weight ratio 1/500.

\*\* Average of two trials.

Reaction temperatures below 50°C were not selected because of the inordinately long reaction time that would be required to obtain complete reaction. Examination of the data in table B-2 indicates the HD/MEA reaction is mildly exothermic and the heat of reaction increases with increasing temperature. A significant change in the heat capacity of the reaction products from temperature of 50° to 65°C was noted. These heat capacity and heat of reaction data tend to support the hypothesis that the products of this reaction change with increasing temperature of reaction.

Rough estimates of the temperature rise in a reaction vessel for a 5:1 volume ratio of MEA:HD, assuming adiabatic conditions, were calculated as a function of reaction temperature (table B-3).

Table B-3. Adiabatic Temperature Rise in the MEA/HD System\*

Initial reaction temperature	Final reaction of system
°C	
50	113
65	151
77	169
86	181

\* These calculations do not take into account the increase in heat of reaction as a function of temperature.

From the data in table B-3, it is apparent that a considerable rise in temperature can occur. In fact, the temperature rise in a reaction vessel at any given temperature will actually be greater than indicated in table B-3 because of the increase of the heat of reaction with increasing temperatures.

### III. CONCLUSION.

MEA is recommended as the decontaminant of choice for the decontamination of K941 and K942 toxic gas sets for the following reasons:

1. MEA is nontoxic, noncorrosive, inexpensive, relatively nonflammable, and has a high boiling point (171°C), which is a desirable property for exothermic reactions.
2. The reaction between MEA and H/HD is relatively fast (half life of 16 minutes at 52°C) with a moderate release of heat.
3. MEA is miscible with H/HD, thus yielding homogeneous reaction conditions.
4. Since MEA and H/HD are required in a ratio of 5:1 without additional solvents, only relatively small quantities of decontamination products have to be transported and/or disposed of.



5. The final decontamination products dissolve in MEA forming a homogeneous solution.

6. The decontamination products are amenable to disposal by incineration, after which gases such as  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{HCl}$ ,  $\text{SO}_x$ , and  $\text{NO}_x$  are formed without leaving any solid residues.

7. The MEA/HD reaction appears to be of a simple nature and does not require sophisticated equipment and can potentially be handled by nonprofessional personnel.

8. The solution containing the decontamination products has been found to meet the Department of Transportation specifications for transport.

Final reaction at 25°C	Final reaction at 50°C
1.1	1.1
1.1	1.1
1.1	1.1
1.1	1.1

## APPENDIX C

### DEMILITARIZATION PROCEDURES

#### I. PREOPERATIONAL PROCEDURES.

The Operations Control Center (OCC) will obtain a daily weather report and a forecast of windspeeds, wind direction, and temperature. Installation personnel will measure the temperature gradient at their own facility. This information will be furnished the installation commander and the officer in charge of the disposal operations. The installation commander will be verbally requested by the OCC to authorize demilitarization operations each day. If permission is granted, the commander will be advised of the estimated completion time. The OCC will alert medical personnel and emergency response teams and all other personnel as dictated by standing operating procedures. The radio nets to the installation control center and medical facility will be tested to assure they are operational prior to the demilitarization operation. Demilitarization personnel will be briefed and dispatched to their respective duties. Once everyone is in place and ready, the OCC will give approval for the operations to begin.

#### II. MOVEMENT PROCEDURES.

The following are the steps necessary for moving the one-ton containers from the storage site to the demilitarization site.

Step 1 - Preparation: All one-ton containers will be visually inspected to assure the valves are not leaking. If a leak is detected, the leak will be sealed and the area decontaminated. All equipment will be inspected to assure proper operation. M8 detection paper (NSN 6665-00-050-8529) will be used to verify contamination and decontamination of the containers and storage area.

Step 2 - Loading: Once it has been ascertained that a one-ton container is safe for movement, the signal will be given to load. Only one one-ton container at a time will be moved to the demilitarization site. All one-ton containers will be escorted to the demilitarization site prior to initiation of demilitarization operations and stored in a selected area at the site. Once the container has been placed on the vehicle, it will be blocked and braced to assure safe transportation.

Step 3 - Movement: After blocking and bracing have been accomplished, the one-ton containers will be escorted in accordance with existing regulations over an approved route to the demilitarization site for downloading.

Step 4 - Storage at Demilitarization Site: In the event the temperature is below 58°F, the one-ton containers will be placed inside a thaw tent at the demilitarization site and temperature will be maintained at approximately 70°F by preheated air fed to the tent. One-ton containers will be reinspected for leakage upon completion of movement and at the beginning of each working day.

### III. OPERATIONAL PROCEDURES.

The following are the steps necessary for the actual demilitarization of the one-ton containers.

1. Figure A-2 illustrates the demilitarization equipment.
2. To initiate operations, the one-ton container is first inspected visually for leaks, and then hoisted by the crane and set onto the one-ton container charging trolley. The one-ton container is oriented in such a way as to position the valves in a vertical line. The one-ton container is then inserted into the one-ton container cabinet by pushing the trolley over its rail line to a metal stop inside the one-ton container cabinet.
3. After insuring that all system valves are closed, the door in the rear of the cabinet is closed and secured.
4. Working through glove ports, the operator, with his assistant, connects the one-ton container to the transfer system. The upper valve is connected first to the line running to the main filter. Next, the lower valve is connected to the line running to the agent transfer system.
5. The reactor is filled with 200 gallons of monoethanolamine (MEA). This is accomplished by the operator's opening the MEA fill valve, agent transfer valves No. 1 and No. 2, and the reactor fill valve. The operator, wearing level C protection with gloves, starts the MEA fill pump and pumps MEA directly to the reactor from the 55-gallon storage drums. The drums are weighed before and after to insure proper filling is achieved. The valves are then completely closed, and the pump is turned off immediately after the fill is completed. All process valves are operated from the outside of the cabinet.
6. A vacuum equivalent to approximately 10 inches of mercury (4.9 psi) is pulled on the agent transfer tank. This is accomplished by starting the vacuum pump, opening the vacuum valve, observing the instrumented pressure reading, and closing the valve and shutting off the pump when the proper vacuum is reached.
7. A 15-gallon increment of mustard is transferred to the agent-transfer tank. This is accomplished by opening the two one-ton container valves and the three agent-transfer valves, causing mustard to fill the agent-transfer tank up to the indicated red view glass line. Then the one-ton container valves and the agent-transfer valves are closed. If the eduction tube of the one-ton container becomes clogged during transfer, the following steps will be taken:
  - a. The agent-transfer valves and one-ton container valves are closed.
  - b. The one-ton container is disconnected from the transfer lines.
  - c. Any mustard in the transfer hose and valve is decontaminated.
  - d. The cabinet is sampled with a blue band tube for vapor contamination.
  - e. The rear cabinet lid is opened.



- f. The one-ton container is extracted and rotated 180 degrees.
- g. The one-ton container is reinserted.
- h. The cabinet is resealed.
- i. The container is connected to the appropriate lines.
- j. The one-ton container and agent-transfer valves are opened.
- k. Transfer to the transfer tank is continued.

8. The agitator and temperature monitoring equipment are started and are given sufficient time to reach a steady operating state. The cooling water through the condensing unit is then started.

9. Fifteen gallons of mustard are charged to the reactor. This is accomplished by first opening the reactor fill valve, then the transfer tank vent valve that vents through the main filter. Upon observing on the view glass that the mustard has been completely drained, the reactor fill valve is closed. Then the transfer tank vent valve is closed.

10. Immediately after charging the reactor with 15 gallons of mustard, the mustard transfer line and the transfer tank are flushed with 25 gallons of MEA to minimize the vapor challenge to the filters. The MEA fill pump is started, and the MEA fill valve and then agent-transfer valves No. 1 and No. 2 are opened. When the MEA has visibly filled the transfer tank to the green 25-gallon fill line, the pump is shut down. All the valves are closed, and the reactor is charged with 25 gallons of MEA by opening the reactor fill valve and the transfer tank vent valve. Then all valves are closed.

11. Continuous temperature monitoring is in effect. When the reaction temperature has reached its peak and begins falling, cooling water is fed to the jacket on the reactor to speed the natural cooling process. To assure the temperature does not exceed 175°F (80°C), the operator and his assistant will turn on the cooling water to the reactor jacket to cool the reaction as necessary to maintain a temperature of 175°F or slightly below. A visible alarm will indicate that cooling water should be fed to the reactor jacket if the temperature exceeds 165°F (74°C). If the temperature should continue to rise, an audible alarm will be activated at 175°F, and the operators will activate the nitrogen introduction system and blanket the reactor with an inert atmosphere. This condition will be maintained until the temperature cools below 175°F.

12. Four successive 15-gallon fills of mustard, each flushed by 25 gallons of MEA, are charged to the reactor as often as cooling and daily schedules permit. There is a total 60 gallons of mustard charged per 300 gallons of MEA per batch. The total reaction and cooling time for each 15-gallon mustard fill is approximately 3 to 5 hours.

13. After four successive charges, the product mix is sampled, analyzed for presence of agent, and then pumped from the reactor to heavy-duty, unlined, 55-gallon drums. If mustard is detected during analysis, MEA will be charged to the reactor in 25-gallon increments until no detectable agent remains using the gas-liquid chromatograph (GLC). If no mustard is detected, the reactor will be emptied by opening the product drain valve and starting the drain pump. The drums

containing the liquid wastes will be stencilled and stored in the vicinity of building E4452. The cooling water and agitator are turned off immediately prior to reactor draining. The temperature monitors run continuously.

14. Once the one-ton container is drained, it is then charged with 50 gallons of MEA. This is accomplished by starting the MEA fill pump and opening the one-ton container valves, MEA fill valve and agent-transfer valve No. 3. Then all valves are closed except the one-ton container valve to the vent. A sufficient reaction time is allowed as monitored by the temperature gauges.

15. Next, the one-ton container is disconnected, removed from the cabinet, rotated 180 degrees and replaced in the cabinet, reconnected to the appropriate lines, and an additional 50 gallons of MEA is introduced as indicated in section 14.

16. After the contents are sampled and certified to be agent free, the one-ton containers will be removed, stencilled, and stored in the vicinity of building E4452. Operations are continued until all eight containers and their contents have been demilitarized.

#### IV. CLEANUP PROCEDURE.

The following are the steps necessary to ensure decontamination of the equipment in preparation for storage:

1. Personnel performing final cleanup will wear level A protective clothing.
2. When the last one-ton container to be demilitarized is removed from the demilitarization cabinet, the MEA fill valve and the agent-transfer valves No. 1 and No. 2 are opened. The reactor fill valve is opened and the MEA fill pump started.
3. The reactor is filled with 500 gallons of MEA, and the agitator is started. Four hours are allowed for any possible reaction. The reactor is then sampled, analyzed (with the GLC), and drained into heavy-duty, unlined, 55-gallon drums. If mustard is detected, four more hours will be allowed and the reactor will be sampled again. This process will be repeated until no detectable mustard remains.
4. The flexible hose connections are removed, placed in an MEA bath for 128 hours, and sampled at eight-hour intervals thereafter until no mustard is detected. Sampling will be accomplished in the same manner as sampling filter core samples with the blue-band detector tube.
5. New hoses will be connected to the apparatus and an empty 15-gallon pail will be placed on the trolley and inserted into the cabinet, which is then resealed.
6. Additional MEA is pumped to the reactor until MEA overflows into the one-ton container vent line. The flexible hose is then removed from the vent line and the line is capped.
7. Additional MEA is pumped to the reactor until the surge tank line overflows into a prepositioned, heavy-duty, unlined, 55-gallon drum. This drum will have been half filled with MEA and the disconnected filter hose placed below the liquid level.

8. The 55-gallon drum, the surge tank, and the reactor will be sampled at eight-hour intervals until no agent is detected. The system will then be drained into heavy-duty, unlined, 55-gallon drums.

9. Additional MEA is pumped to the 15-gallon pail placed on the one-ton container trolley by opening the MEA fill valve and agent-transfer valve No. 3. The remaining flexible hose is then disconnected, placed in the pail, and the line is capped.

10. After 128 hours, the 15-gallon pail is removed and sampled. When decontamination is assured, the MEA is placed in a heavy-duty, unlined, 55-gallon drum, the hoses are disposed of as solid waste, and the pail is replaced on the trolley and sealed in the cabinet.

11. The reactor vent line is capped.

12. The vacuum filters are unpacked and disposed of by soaking for 128 hours in MEA, sampling for residual contamination, and repeated soaking if necessary. They are then double wrapped in plastic containers and shipped to Edgewood Arsenal for final disposal.

13. The main filters are unpacked and disposed of as described in section IV.12.

14. The MEA shower system is activated and the chamber is washed with 200 gallons of MEA. After 128 hours, the waste is sampled at eight-hour intervals and drained when decontaminated. The cabinet is then additionally sampled using the blue-band tube.

15. The cabinet filters are unpacked and disposed of as described in section IV.12. The cabinet vent is then capped.

16. The utilities are disconnected from the demilitarization cabinet.

17. The area surrounding the demilitarization apparatus, the thaw tent, and all vehicles will be checked for contamination and decontaminated as necessary.

18. All solid wastes such as cloths, syringes, and blue-band tubes are decontaminated and disposed of by landfill.

19. The hot line will be disbanded.

20. All 55-gallon metal drums and one-ton containers containing decontaminated wastes will be stencilled as follows: "Neutralized Products, HD-MEA" and "DATE \_\_\_\_\_" and stored temporarily in the chemical agent storage yard at Fort McClellan. Drums containing liquid supertropical bleach (STB) wastes will be stencilled "Neutralized Products, HD-STB" and "DATE \_\_\_\_\_" and also retained.

21. All unused supplies will be returned to storage. All demilitarization equipment used in the operation will be shipped to Anniston Army Depot. None of the equipment will be used on other projects unless approved by the US Army Materiel Command Program Manager for Demilitarization of Chemical Materiel.



## V. ULTIMATE WASTE DISPOSAL.

### A. Description of Waste.

The demilitarized waste will be drained from the reactor into heavy-duty, unlined, 55-gallon drums. It is anticipated that approximately 250 drums of waste will be generated by the operation, each holding approximately 50 gallons. In addition, there will be eight detoxified one-ton containers holding approximately 100 gallons of waste each.

### B. Shipment of Waste.

The demilitarized waste is *not* to be retained at Fort McClellan. The drums are to be labeled "Corrosive" (in addition to stencil and other appropriate DOT labels) immediately following the close of operations and are also to be palletized in preparation for shipment by Army or commercial truck. The one-ton containers will be labeled identically. As soon as labeling and packaging operations are completed, the waste containers are to be sent to Rocky Mountain Arsenal (RMA), Denver, Colorado, for ultimate disposal. All labeling and packaging operations will be the responsibility of Fort McClellan personnel. Arrangement for shipment will be made by Edgewood Arsenal with loading operations conducted by Fort McClellan personnel. Unloading operations will be conducted by RMA personnel.

### C. Incineration of Waste.

The demilitarized waste is *not* to be retained in storage at RMA. As soon as is feasible following the arrival of the waste, incineration operations will begin.

Tests have been conducted both at Edgewood Arsenal and at RMA to insure that pollution of the atmosphere by  $\text{SO}_2$ , particulates, or  $\text{NO}_x$  does not occur. Copies of these test reports, which include a one-hour test burn in the RMA furnace, are in appendix F.

Personnel from RMA will utilize available plant equipment to drain the liquid from both the 55-gallon drums and the one-ton containers for incineration in the bulk liquid of the hydrazine furnace formerly used to incinerate mustard agent (project EAGLE, phase I). As the containers are emptied, they will be fed to the two one-ton container furnaces and incinerated under procedures used for incinerating agent-filled containers during project EAGLE, phase I. Personnel involved in the draining operation will wear a minimum of level C protective clothing (with gloves).

The resulting residue from the incineration operation will consist of inert salts identical to those obtained from the incineration of mustard at RMA, and of scrap iron. The inert salts will be disposed of in an environmentally acceptable manner. The metal containers will be sold as scrap to private industry.

## APPENDIX D

### DEPARTMENT OF TRANSPORTATION (DOT) DOCUMENTATION

This section is included to provide laboratory documentation for the liquid waste to be generated by the demilitarization operation.

The following data are provided in order: (1) Results of toxicity testing on two pilot runs made with mustard (HD) and monoethanolamine (MEA); (2) results of toxicity testing on a pilot run made with mustard (H) and MEA; and (3) raw data (tables D-1 through D-6) from DOT tests conducted in the Biomedical Laboratory, Edgewood Arsenal, Maryland.

#### 1. Toxicity Testing on Pilot Runs.<sup>1</sup>

The following samples were tested in accordance with CFR49, parts 100 to 119, revised as of 1 January 1972:

a. HD/MEA — Run No. 2 (supplied by P. Mirabella), received 8 January 1974.

b. HD/MEA — Run No. 4 (supplied by P. Mirabella), received 30 March 1974.

These samples were shown to be less toxic orally, by inhalation, and by skin absorption than the standards cited in section 173.343, CFR49: "Less dangerous poisons, class B, liquid or solid, poison label; definition."

#### 2. Toxicity Testing on Pilot Runs.<sup>2</sup>

The following samples were tested in accordance with CFR49, parts 100 to 119, revised 1 January 1972:

a. CG/NaOH — run No. 14. Sample received 4 October 1974 from P. Mirabella.

b. H(Levenstein mustard)/MEA — run No. 18. Sample received 18 October 1974 from P. Mirabella.

The tested samples were shown to be less toxic orally, by inhalation, and by skin absorption than the standards cited in section 173.343, CFR49: "Less dangerous poisons, class B, liquid or solid, poison label; definition."

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<sup>1</sup>Owens, Edmund. CFR49 Class B Poison Evaluation. Biomedical Laboratories, Edgewood Arsenal. 30 October 1974.

<sup>2</sup>Owens, Edmund. CFR49 Class B Poison Evaluation. Biomedical Laboratories, Edgewood Arsenal. 5 November 1974.

Table D-1. Results of CFR 49 (DOT) Tests on Obsolete War Gas Set Residues Received from Disposal Engineering Division, Manufacturing Technology Directorate

Work package item nomenclature	Sample designation	Sample number	Date received	48-Hour mortality				Remarks
				Oral – rat (50 mg/kg)	Skin – rabbit (200 mg/kg)	Inhalation – rat		
						(2 mg/l – 1 hr)	(>2 mg, <200 mg/l – 1 hr)	
No. 1 chemical reactor system	HD/MEA	GID 1	11/21/73	8/10	a	a	a	a
	HD/MEA	GID 2	8/01/74	0/10	0/10	0/10	-	Passed DOT screen <sup>b</sup>
	HD/MEA	GID 4	4/30/74	0/10	0/10	0/10	-	Passed DOT screen
	CG/NaOH	GID 5	9/16/74	1/10	0/10	0/10	-	Passed DOT screen
Work package con- trol number 5-1-8, expenditure order 5S05-BB	CG/NaOH	GID 6	6/26/74	0/10	0/10	0/10	-	Passed DOT screen
	HD/CHCl <sub>3</sub> /MEA	GID 7	7/23/74	2/10	0/10 <sup>c</sup>	0/10	-	Passed DOT screen
Samples furnished by P. Mirabella	4 CHCl <sub>3</sub> /NaOH	GID 9	8/19/74	0/10	0/10	0/10	-	Passed DOT screen
	50% PS/CHCl <sub>3</sub> /MEA	GID 10	8/19/74	0/10	0/10	0/10	-	Passed DOT screen
	HD/CHCl <sub>3</sub> /MEA	GID 13	9/27/74	0/10	0/10	0/10	-	Passed DOT screen
	CG/NaOH	GID 14	10/04/74	0/10	3/10	0/10	-	Passed DOT screen
	L/CHCl <sub>3</sub> /NaOH	GID 15	10/08/74	0/10	1/10	0/10	-	Passed DOT screen
	CK/NaOH	GID 16	10/08/74	0/10	0/10	0/10	-	Passed DOT screen
	HN-1/MEA	GID 17-7	10/18/74	0/10	0/10	0/10	-	Passed DOT screen
	H(Levenstein)/MEA	GID 18-4	10/18/74	0/10	0/10	0/10	-	Passed DOT screen
	CHCl <sub>3</sub> /MEA (control)	-	9/12/74	0/10	0/10	0/10	-	Passed DOT screen
	CG/NaOH	GID 19	10/29/74	0/10	0/10	0/10	-	Passed DOT screen
	CK/NaOH	GID 20	10/31/74	0/10	0/10	0/10	-	Passed DOT screen
	H/CHCl <sub>3</sub> /NaOH	GID 21 <sup>d</sup>	11/18/74	0/10	0/10	0/10	0/10	Passed DOT screen
	HN-1/CHCl <sub>3</sub> /MEA	GID 22 <sup>d</sup>	11/21/74	0/10	2/10	0/10	0/10	Passed DOT screen

NOTE: See footnotes at end of table.



Table D-1. Continued

Work package item nomenclature	Sample designation	Sample number	Date received	48-Hour mortality				Remarks
				Oral - rat (50 mg/kg)	Skin - rabbit (200 mg/kg)	Inhalation - rat		
						(2 mg/l - 1 hr)	(>2 mg, <200 mg/l - 1 hr)	
No. 1 chemical reactor system. Work package control No. 5-1-8, expenditure order 5S05-BB	CK/NaOH	GID 23 <sup>d</sup>	11/25/74	0/10	2/10	0/10	0/10	Passed DOT screen
	PS/CHCl <sub>3</sub> /MEA	10-A-3 <sup>d</sup>	1/06/75	0/10	1/10	0/10	0/10	Passed DOT screen
	HD/CHCl <sub>3</sub> /MEA	GID 24 <sup>d</sup>	1/08/75	0/10	0/10	0/10	0/10	Passed DOT screen
Samples furnished by P. Mirabella	PS/CHCl <sub>3</sub> /MEA	GID 25 <sup>d</sup>	1/02/75	0/10	0/10	0/10	0/10	Passed DOT screen
	CG waste	GID 26 <sup>d</sup>	1/16/75	0/10	1/10	0/10	0/10	Passed DOT screen
	CG waste	GID 27 <sup>d</sup>	1/16/75	0/10	0/10	0/10	0/10	Passed DOT screen
	CK demilitarization product	GID 29 <sup>d</sup>	1/24/75	0/10	0/10	0/10	0/10	Passed DOT screen
Sample furnished by Disposal Engineer- ing Division - no source shown. Tested under expenditure order 5S05-BB or 5S05-01	Levenstein mustard 5:1 v/v 31-9115-52	-	9/21/74	0/10	1/10	0/10	-	Passed DOT screen
	HD in CHCl <sub>3</sub> (1 part) MEA (2 parts) sample No. 20	-	1/10/74	1/10	0/10 <sup>c</sup>	0/10	-	-
	HD destruction No. 1; 75 gm methyl alcohol 75 gm 20% aqueous NaOH 40 gm HD neutralized with H <sub>2</sub> SO <sub>4</sub>	-	4/09/74	0/10	0/10	0/10	-	-
Additional samples furnished - all marked Garono - plus sample designation. Tested under expenditure order No. 5S05-BB or 5S05-01	HD destruction No. 2; 1.5 gm triton x100 150 gm 20% aqueous NaOH 40 gm HD	-	4/09/74	0/10	0/10	0/10	-	Passed DOT screen

NOTE: See footnotes at end of table.

Table D-1. Continued

Work package item nomenclature	Sample designation	Sample number	Date received	48-Hour mortality				Remarks
				Oral - rat (50 mg/kg)	Skin - rabbit (200 mg/kg)	(2 mg/l - 1 hr)	Inhalation - rat > 2 mg, <200 mg/l - 1 hr	
Additional samples furnished - all marked Garono - plus sample designation. Tested under expenditure order No. SS05-BB or SS05-01 (Contd)	HD destruction No. 4; 75 gm 20% aqueous NaOH 75 gm methyl cellulose 40 gm HD	-	4/09/74	0/10	0/10	0/10	-	Passed DOT screen
	Methyl cellulose	-	4/09/74	0/10	0/10	0/10	-	Passed DOT screen
	5 gm HD in 5% NaOH neutralized with 18N H <sub>2</sub> SO <sub>4</sub> . 4/24/74. Run HD-1	-	5/02/74	0/10	0/10	0/10	-	Passed DOT screen
Additional samples furnished - all marked Garono - plus sample designation. Tested under expenditure order SS05-BB or SS03-01	10 gm HD in 10% NaOH neutralized with 18N H <sub>2</sub> SO <sub>4</sub> . 4/24/74. Run HD-1	-	5/02/74	0/10	0/10	0/10	-	Passed DOT screen
	20 gm HD in 20% NaOH neutralized with 18N H <sub>2</sub> SO <sub>4</sub> . 4/24/74. Run HD-1	-	5/02/74	0/10	0/10	0/10	-	Passed DOT screen
	5.0 cc each 5% HD in CHCl <sub>3</sub> . 5% L in CHCl <sub>3</sub> decontaminated with 5% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/28/74-6/4/74)	-	6/05/74	0/10	0/10	0/10	-	Passed DOT screen
	10.0 cc each 5% HD in CHCl <sub>3</sub> . 5% L in CHCl <sub>3</sub> decontaminated with 10% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/25/74-6/4/74)	-	6/05/74	0/10	0/10	0/10	-	Passed DOT screen

Table D-1. Continued

Work package item nomenclature	Sample designation	Sample number	Date received	48-Hour mortality			Remarks	
				Oral - rat (50 mg/kg)	Skin - rabbit (200 mg/kg)	Inhalation - rat		
						(2 mg/l - 1 hr)		(>2 mg, <200 mg/l - 1 hr)
Additional samples furnished - all marked Garono - plus sample designation. Tested under expenditure order SS05-BB or SS03-01 (Contd)	20 cc each 5% HD in CHCl <sub>3</sub> , 5% L in CHCl <sub>3</sub> decontaminated with 20% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/28/74-6/4/74)	-	6/05/74	0/10	0/10	-	Passed DOT screen	
	HD in CHCl <sub>3</sub> decontami- nated with 5% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/8/74-6/4/74)	-	6/05/74	0/10	0/10	-	Passed DOT screen	
	5% HD in CHCl <sub>3</sub> decon- taminated with 10% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/8/74-6/4/74)	-	6/05/74	0/10	0/10	-	Passed DOT screen	
	5% HD in CHCl <sub>3</sub> decon- taminated with 20% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/8/74-6/4/74)	-	6/05/74	0/10	0/10	e	Passed DOT screen	
	5 gm Lewisite in 5% NaOH neutralized with 18N H <sub>2</sub> SO <sub>4</sub> (4/29/74) Run L-1	-	5/02/74	0/10	0/10	0/10	-	Passed DOT screen
	10 gm Lewisite in 10% NaOH neutralized with 18N H <sub>2</sub> SO <sub>4</sub> (4/29/74) Run L-1	-	5/02/74	0/10	0/10	0/10	-	Passed DOT screen
Additional samples furnished - all marked Garono - plus sample designation. Tested under expenditure order SS05-BB or SD05-01								

NOTE: See footnotes at end of table.



Table D-1. Continued

Work package item nomenclature	Sample designation	Sample number	Date received	48-Hour mortality				Remarks
				Oral - rat (50 mg/kg)	Skin - rabbit (200 mg/kg)	(2 mg/l - 1 hr)	Inhalation - rat (>2 mg, <200 mg/l - 1 hr)	
Additional samples furnished - all marked Garono - plus sample designation. Tested under expenditure order 5S05-BB or 5D05-01 (Contd)	20 gm Lewisite in 20% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (2/29/74) Run L-1	-	5/02/74	0/10	1/10	0/10	-	Passed DOT screen
	50% PS decontaminated with 5% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/13/74-6/4/74)	-	6/05/74	0/10	0/10	0/10	-	Passed DOT screen
	50% PS decontaminated with 10% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/13/74-6/4/74)	-	6/05/74	0/10	0/10	0/10	-	Passed DOT screen
	50% PS decontaminated with 20% NaOH neutralized with H <sub>2</sub> SO <sub>4</sub> (5/13/74-6/4/74)	-	6/05/74	0/10	0/10	0/10	-	Passed DOT screen

<sup>a</sup> Tests not completed - failed oral test, CFR 40.<sup>b</sup> CFR 49 status (p section 173.343) - passed DOT screen, not a class B poison.<sup>c</sup> Eschar formation.<sup>d</sup> Secondary tests initiated on these samples - skin, eye, intravenous, and oral testing not requested.<sup>e</sup> Insufficient sample.

Table D-2. Results of Intravenous and Oral LD50 Determinations (Secondary Tests) on Obsolete War Gas Set Residues from Disposal Engineering Division, Manufacturing Technology Directorate<sup>a</sup>

Work package item nomenclature	Sample designation	Sample number	Date received	Intravenous LD50 <sup>b</sup> (mouse)	Oral LD50 <sup>b</sup> (rat)	Toxicity class	
						iv - mouse <sup>c</sup>	Oral - rat <sup>d</sup>
No. 1 chemical reactor system	HD/MEA	GID 2	1/08/74	1.2 (1.0-1.4)	0.43 (0.26-0.71)	5	4
	HD/MEA	GID 4	4/30/74	0.49 (0.41-0.57)	1.52 (1.26-1.83)	4	4
	50% PS/CHCl <sub>3</sub> /MEA	GID 10	8/19/74	0.30 (0.26-0.33)	1.16 (0.91-1.47)	4	4
	HD/CHCl <sub>3</sub> /MEA	GID 13	9/27/74	0.45 (0.41-0.51)	1.81 (1.51-2.18)	4	4
Work package control No. 5-1-8 Expenditure order 5S05-BB	CG/NaOH	GID 14	10/04/74	0.92 (0.76-1.10)	2.62 (1.78-3.80)	5	5
	L/CHCl <sub>3</sub> /NaOH	GID 15	10/08/74	0.82 (0.56-1.25)	2.11 (1.71-2.60)	4	4
	CK/NaOH	GID 16	10/08/74	0.46 (0.40-0.53)	2.32 (1.92-2.80)	4	4
	HN-1/MEA	GID 17-7	10/18/74	0.44 (0.37-0.53)	1.47 (1.34-1.62)	4	4
	H(Levenstein)/MEA	GID 18-4	10/18/74	0.44 (0.39-0.59)	1.73 (1.53-1.95)	4	4
	CHCl <sub>3</sub> /MEA (control)	-	9/12/74	0.43 (0.39-0.47)	1.48 (1.12-1.97)	4	4

<sup>a</sup> Hodge, H. E., and Sterner, J. H. Combined Tabulation of Toxicity Classes. American Industrial Hygiene Association Quarterly 10:4, 93, December 1943.

<sup>b</sup> Range shown in parentheses represents 95% confidence limits; data represents 14-day observation.

<sup>c</sup> 4 = slightly toxic, for range of 0.05 to 0.5 gm/kg; 5 = practically nontoxic, for range of 0.5 to 1.5 gm/kg.

<sup>d</sup> 4 = slightly toxic, for range of 0.5 to 5.0 gm/kg; 5 = practically nontoxic, for range of 5 to 15 gm/kg.

Table D-3. Results of CFR 21\* Secondary Tests (Oral, Skin, and Inhalation Mortality) on Obsolete War Gas Set Residues from Disposal Engineering Division, Manufacturing Technology Directorate  
(Work package designation, No. 1 chemical reactor system, work package control No. 5-1-8 - expenditure order No. 6S05-BB)

Sample designation	Sample number	Date received	Toxicity and classification - 14-day observation											
			Oral - rat			Skin - rabbit (24-hour contact)			Inhalation - rat (1-hour exposure)					
			50 mg/kg		5.0 gm/kg	200 mg/kg		2.0 gm/kg	<2.0 mg/l		> 2 < 200 mg/l			
			Mortality	Class	Mortality	Mortality	Class	Mortality	Mortality	Class	Mortality	Class	Mortality	Class
HD/MEA	GID 2	1/08/74	0/10	N	10/10	T	0/10	N	0/10	<T	0/10	N	0/10	N
HD/MEA	GID 4	4/30/74	0/10	N	10/10	T	0/10	N	0/10	<T	0/10	N	0/10	N
50% PS/CHCl <sub>3</sub> /MEA	GID 10	8/19/74	0/10	N	10/10	T	1/10	<HT	0/10	<T	0/10	N	0/10	N
HD/CHCl <sub>3</sub> /MEA	GID 13	9/27/74	0/10	N	10/10	T	2/10	<HT	0/10	T	0/10	N	0/10	N
CG/NaOH	GID 14	10/04/74	0/10	N	7/10	T	0/10	N	0/10	N	0/10	N	0/10	N
L/CHCl <sub>3</sub> /NaOH	GID 15	10/08/74	0/10	N	10/10	T	1/10	<HT	0/10	<T	0/10	N	0/10	N
CH/NaOH	GID 16	10/08/74	0/10	N	9/10	T	0/10	N	0/10	<T	0/10	N	0/10	N
HN-1/MEA	GID 17	10/18/74	0/10	N	10/10	T	0/10	N	0/10	N	0/10	N	0/10	N
H(Levenstein)/MEA	GID 18-4	10/08/74	0/10	N	10/10	T	0/10	N	0/10	N	0/10	N	0/10	N
CHCl <sub>3</sub> /MEA (control)	-	9/12/74	0/10	N	10/10	T	2/10	<HT	0/10	N	0/10	N	0/10	N
CG/NaOH	GID 19	10/29/74	0/10	N	-	-	-	-	0/10	-	0/10	N	0/10	N
CH/NaOH	GID 20	10/31/74	0/10	N	-	-	-	-	0/10	-	0/10	N	0/10	N

\* CFR 21 toxicity classification of "Hazardous Substances" (p section 191.1).

NOTES: N = negative; T = toxic; <T = less than toxic classification; HT = highly toxic; <HT = less than highly toxic classification.



Table D-4. Results of CFR 21<sup>a</sup> Secondary Tests (Eye and Skin Irritation) on Obsolete War Gas Set Residues  
Received from Disposal Engineering Division, Manufacturing Technology Directorate

(Work package item nomenclature, No. 1 chemical reactor system, work package control No. 5-1-8, expenditure order No. 5S05-BB)

Sample designation	Sample number	Date received	Irritancy tests and classifications											
			Eye <sup>b</sup> - rabbit (ml/eye)						Skin <sup>c</sup> - rabbit (ml/back) - intact and abraded					
			0.1		0.01		0.001		0.5		0.05		0.005	
			Response fraction	Class	Response fraction	Class	Response fraction	Class	Response fraction	Class	Response fraction	Class	Response fraction	Class
HD/MEA	GID 2	1/08/74	6/6	POI	4/6	POI	0/6	N	4.4	<PRI	0.6	<PRI	0.1	<PRI
HD/MEA	GID 4	4/30/74	6/6	POI	5/6	POI	0/6	N	4.7	<PRI	3.9	<PRI	1.3	<PRI
50% PS/CHCl <sub>3</sub> /MEA	GID 10	8/19/74	6/6	POI	6/6	POI	0/6	N	6.0	PRI	3.5	<PRI	1.7	<PRI
HD/CHCl <sub>3</sub> /MEA	GID 13	9/27/74	6/6	POI	6/6	POI	0/6	N	4.5	<PRI	2.4	<PRI	0.2	<PRI
CG/NaOH	GID 14	10/04/74	6/6	POI	6/6	POI	0/6	N	0	N	0	N	0	N
L/CHCl <sub>3</sub> /NaOH	GID 15	10/08/74	6/6	POI	6/6	POI	1/6	<POI	1.4	<PRI	2.7	<PRI	0.5	<PRI
CK/NaOH	GID 16	10/08/74	6/6	POI	6/6	POI	4/6	POI	2.0	<PRI	1.0	<PRI	0	N
HN-1/MEA	GID 17-7	10/18/74	6/6	POI	6/6	POI	0/6	N	5.5	PRI	4.3	<PRI	0.8	<PRI
H(Levenstein)/MEA	GID 18-4	10/18/74	6/6	POI	6/6	POI	6/6	N	5.5	PRI	3.9	<PRI	3.0	<PRI
CHCl <sub>3</sub> /MEA - Control	-	9/12/74	6/6	POI	6/6	POI	0/6	N	6.3	PRI	3.0	<PRI	0.8	<PRI

<sup>a</sup> CFR 21 classification "Scoring Irritants" (p sections 191.1-191.10, 191.11, 191.12).

<sup>b</sup> Eye - 0.1 mg/eye -

The test shall be considered positive if four or more of the animals in the test group exhibit a positive reaction. If only one animal exhibits a positive reaction, the test shall be regarded as a negative. If two or three animals exhibit a positive reaction, the test is repeated using a different group of six animals. The second test shall be considered positive if three or more of the animals exhibit a positive reaction. If only one or two animals in the second test exhibit a positive reaction, the test shall be repeated with a different group of six animals. Should a third test be needed, the substance will be regarded as an irritant if any animal exhibits a positive response.

<sup>c</sup> Skin -

The term "primary irritant" means a substance that is not corrosive and that the available data of human experience indicate is a primary irritant; or which results in an empirical score of five or more when tested by the method described in section 191.11.

NOTE: N = negative test; POI = positive eye irritant; < POI = less than positive irritant class; PRI = primary irritant; and < PRI = less than positive irritant class.

Table D-5. Results of Aquatic Species Tests (Secondary) on Obsolete War Gas Set Residues Received from Chemical Laboratory<sup>a</sup>

Sample designation	Sample number	26-Hour TL 50 (95% confidence limits)					
		Fathead minnow		Bluegill		Mosquito larvae	
		ppm <sup>b</sup>	Toxicity rating	ppm <sup>b</sup>	Toxicity rating	ppm <sup>b</sup>	Toxicity rating <sup>c</sup>
HD/MEA	GID 2	500 (138-1805)	1	241 (170-341)	1	>1000	0
HD/MEA	GID 4	750 (364-1546)	1	414 (337-509)	1	>1000	0
PS/CHCl <sub>3</sub> /MEA	GID 10	319 (291-350)	1	302 (273-336)	1	>1000	0
HD/CHCl <sub>3</sub> /MEA	GID 13	491 (456-527)	1	416 (360-480)	1	>1000	0
CG/NaOH	GID 14	669 (615-727)	1	528 (440-634)	1	>1000	0
L/CHCl <sub>3</sub> /NaOH	GID 15	461 (408-521)	1	596 (565-628)	1	840 (527-1337)	1
CH/NaOH	GID 16	318 (276-364)	1	168 (151-187)	1	>1000	0
HN-1/MEA	GID 17-7	561 (467-674)	1	384 (346-427)	1	>1000	0
H(Levenstein)/MEA	GID 18-4	323 (284-355)	1	255 (211-307)	1	>1000	0
CHCl <sub>3</sub> /MEA (control)	-	-	-	218 (124-386)	1	>1000	0

<sup>a</sup>Ham, R. W., Jr., and Jensen, R. A. Water Quality Characteristics of Hazardous Material. Environmental Engineering Division, Civil Engineering Department, Texas A&M University. Evaluation of the Hazard of Bulk Water Transportation of Industrial Chemicals. A Tentative Guide. National Academy of Sciences, National Research Council, Washington, D.C. 1966.

<sup>b</sup>Range shown in parentheses represents 95% confidence limits.

<sup>c</sup>0 = insignificant hazard, for range >1000 ppm; 1 = practically nontoxic, for range 100 to 1000 ppm.

Table D-6. Results of CFR 49 (DOT) Tests on Obsolete War Gas Set Residues from Disposal Engineering Division, Manufacturing Technology Directorate<sup>a</sup>

Sample designation	Sample number	Date received	48-Hour mortality			Remarks
			Oral - rat <sup>b</sup>	Skin - rabbit <sup>c</sup>	Inhalation - rat <sup>d</sup>	
MEA/CK (10:1 v/v)	32-8661-106	12/02/74	0/10	0/10	0/10	Passed all tests, not class B poison (CFR 49 p section 173.343)
MEA/CG (10:1 v/v)	32-8661-107	12/02/74	0/10	0/10	0/10	Passed all tests, not class B poison (CFR 49 p section 173.343)
L/MEA	32-2614-146	11/12/74	0/10	0/10	0/10	Passed all tests, not class B poison (CFR 49 p section 173.343)
MEA/HD (10:1 v/v)	34-9115-118	12/17/74	0/10	0/10	0/10	Passed all tests, not class B poison (CFR 49 p section 173.343)
MEA/(5% HD/CHCl <sub>3</sub> ; 10% HN-1/CHCl <sub>3</sub> ; 5% L/CHCl <sub>3</sub> ; .5% PS/CHCl <sub>3</sub> ; Neat CK/Neat CG) - 12.5 v/v	35-9115-119	12/17/74	0/10	0/10	0/10	Passed all tests, not class B poison (CFR 49 p section 173.343)

<sup>a</sup>Work package item nomenclature - No. 5 dunnage, work package control No. 5-5-8, expenditure order No. 5S05-KP.

<sup>b</sup>Dosage = 50 mg/kg.

<sup>c</sup>Dosage = 200 mg/kg.

<sup>d</sup>Dosage = 2 mg/l for 1 hour.



## APPENDIX E

### PILOT TEST DATA

The process of incrementally demilitarizing mustard in monoethanolamine (MEA) was successfully piloted by the demilitarization of obsolete toxic gas sets program. Results of one such pilot run closely simulating Fort McClellan operating conditions on a smaller scale are included in this appendix.

The gas identification (GID) sets run plan, HD disposal run No. 4, as of 16 April 1974 consisted of the following sequence of steps:

1. Thirty-five gallons of MEA will be charged to drum (296 pounds). MEA temperature will be between 20° and 25°C before adding distilled mustard (HD).
2. Seven gallons of HD will be destroyed in two days. Three and one-half gallons of HD (37 pounds) will be added on the first day at a rate equivalent to six bottles per minute (1.76 lb/min for 21 minutes).
3. A control vapor sample will be collected before addition of HD. Repetitive vapor samples will be collected during addition of HD and subsequent reaction. Vapor samples will be analyzed from the filter exhaust by a hyfed analyzer.
4. The reaction of HD and MEA will proceed without auxiliary cooling. The solution temperature will be recorded and allowed to reach its peak. Data will be collected while the solution cools slowly.
5. The second half of the HD will be added to the partially used MEA solution on the second day with the MEA cooled to between 20° and 25°C. Feed rate will be the same as during the first batch.
6. The solution temperature will again be allowed to rise and data will be collected.
7. At the conclusion of the second day, two liquid samples will be drawn. A 20-ml analytical sample will be frozen until it can be delivered for analysis. A 3-liter sample will be collected for Department of Transportation (DOT) testing.

Table E-1 is the laboratory record of the GID Run No. 4 HD/MEA reaction temperature history. Figure E-1 is the corresponding graph of temperature (°C) versus elapsed time for the same reaction run.

Table E-1. Distilled Mustard (HD) Run No. 4

Clock time	Elapsed time		Temperature	Comments
	hr	min	°C	
<u>18 April 1974</u>				
1332	0	0	22.5	HD started into drum
1352	0	20	26	HD addition completed
1415	0	43	30	
1430	0	58	34	
1500	1	28	53.5	
1515	1	43	85	Maximum temperature (1 hr, 43 min from start of HD addition)
1524	1	52	85	Temperature starting to drop
1536	2	04	83	
1630	2	58	76	
1700	3	28	72.5	
1800	4	28	66	
1900	5	28	61	
2000	6	28	56	Mixer off
<u>19 April 1974</u>				
0835	19	03	33	
0918	19	46	33	Cooling started (tapwater)
1013	20	41	23	Cooling stopped
1018	20	46	23	HD addition started
1040	21	08	24	HD addition complete
1121	21	49	33	
1145	22	13	41	
1200	22	28	56	
1215	22	43	77	Maximum temperature (1 hr, 57 min from start of HD addition)
1230	22	58	77	Temperature starting to drop
1245	23	13	75	
1300	23	28	73	
1330	23	58	70	
1405	24	33	66	
1501	25	29	60	
1533	26	01	58	
1630	26	58	54	
1815	28	43	48	
1830	18	58	47	
2015	30	43	43	
2025	30	53	43	Run complete

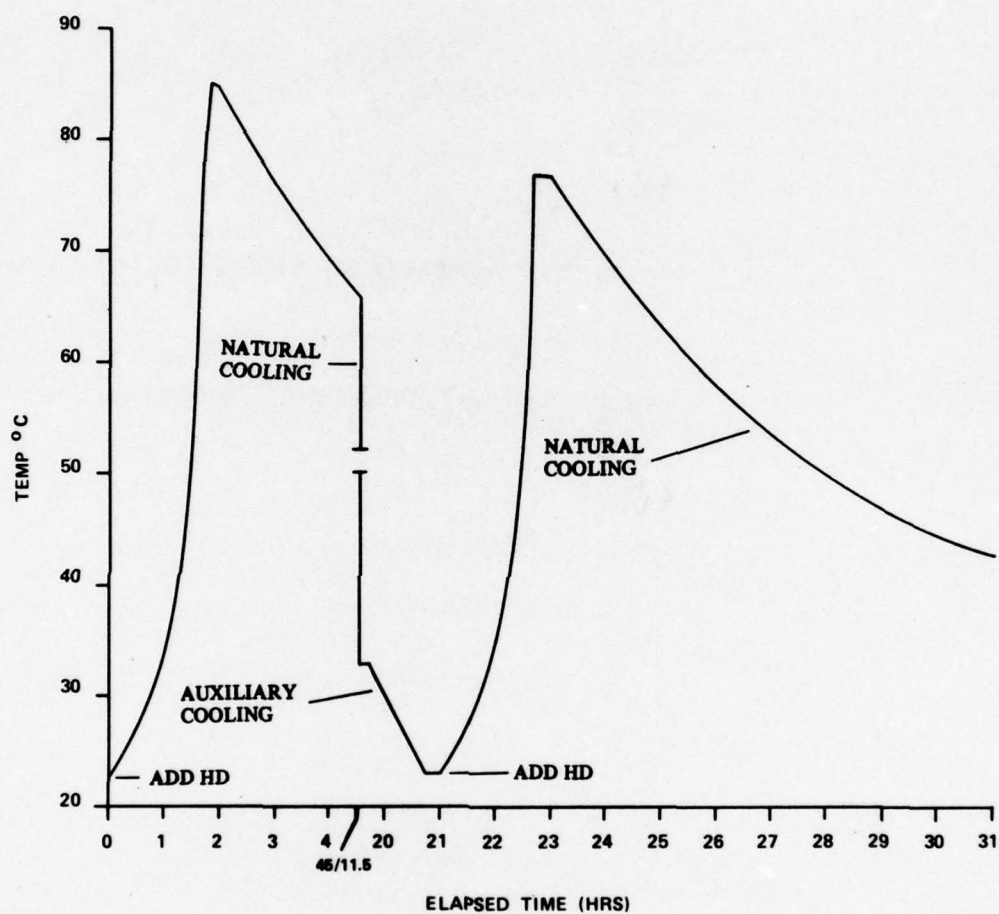


Figure E-1. Temperature Versus Time – Distilled Mustard (HD) in Monoethanolamine (MEA)



## APPENDIX F

### WASTE INCINERATION STUDIES

This section is provided to document the statement that the waste liquid can be safely incinerated and converted to inert salts with no detrimental environmental effect. The following documentation is provided:

1. Incineration Studies of a Solution of Detoxified Mustard (HD)—Monoethanolamine (MEA) Wastes (Edgewood Arsenal).
2. Results of Incineration Testing at Rocky Mountain Arsenal (RMA).
3. Test Plan — Determination of Feasibility of Incinerating Monoethanolamine (MEA) Waste from Fort McClellan, Alabama.
4. Results of Air Pollution Source Survey at Rocky Mountain Arsenal by the US Army Environmental Hygiene Agency.

#### 1. Incineration Studies of a Solution of Detoxified Mustard (HD)—Monoethanolamine (MEA) Waste.<sup>1</sup>

Due to the prohibition of the shipping of toxic chemicals such as mustard (HD), it has been proposed that HD be detoxified onsite by reaction with monoethanolamine (MEA) and the resulting solution then be shipped to an existing incinerator. Experiments were requested to determine on a laboratory scale if there were any problems to be encountered in the incineration of an HD-MEA solution. Also requested was the recovery of hydrogen chloride, recovery and ratio of sulfur dioxide ( $\text{SO}_2$ ) and sulfur trioxide ( $\text{SO}_3$ ), and the amount of  $\text{NO}_x$  formed.

A sample of the HD-MEA waste was submitted to the Analytical Chemistry Branch by the Manufacturing Technology Directorate, Edgewood Arsenal, for these incineration studies.

Six portions of the sample submitted, gas identification sets (GID Run No. 4, 18 April 1974, HD-MEA, from drum 10-18-74, were subjected to incineration in the laboratory furnace described in EATR 4516.<sup>2</sup> No problems were observed in the liquid feed, incineration, or effluent scrubber sections of the process. Nothing was observed to indicate that there might be any potential problems in incinerating this type of solution. The incineration did not appear to generate as much heat as a neat HD incineration. The elemental analysis of the HD-MEA as received was 38.8% carbon, 10.3% hydrogen, 17.8% nitrogen, 4.4% sulfur, and 7.9% chlorine. The density was 1.11 gm/ml.

Data on the incineration runs are summarized in table F-1. Average data from EATR 4516<sup>2</sup> are included in table F-1 for comparison. Recovery in the effluent appears comparable to that obtained in previous work; 92% to 97% of the chlorine and 95% to 99% of the sulfur were collected. The only difference noted was that the conversion of detoxified HD to  $\text{SO}_2$  during incineration was approximately 5% less than that obtained in neat HD incinerations. Whether this is significant is not evident at this time.  $\text{NO}_2$  in the effluent was less than the minimum detection level of 25  $\mu\text{g}$  in each run.

This completes the incineration and analyses requested on this sample.

Table F-1. Incineration Data

Run No.	Total input gm	Cl <sup>-</sup> recovered		Sulfur recovered				NO <sup>**</sup> <sub>x</sub>	Furnace temperature °C
				Total*		As SO <sub>2</sub>			
						gm	%		
1	11.15	0.818	92.74	0.493	99.80	0.245	49.60	—	750
2	10.59	0.766	91.52	0.462	98.50	0.372	79.32	—	800
3	11.15	0.850	96.37	0.478	96.76	0.368	74.49	7.8	800
4	11.15	0.847	96.03	0.485	98.18	0.391	79.15	15.6	800
5	11.15	0.851	96.49	0.472	95.55	0.370	74.90	14.1	800
6	11.15	0.857	97.17	0.470	95.14	0.363	73.48	8.0	800
Average from EATR 4516	13.1	—	92-97	—	92-97	—	79-88	—	800

\* Sulfur recovered as SO<sub>3</sub> is calculated by the difference between the total sulfur and the sulfur recovered as SO<sub>2</sub>.

\*\* The oxygen (as air) supplied was 117% of the theoretical amount required for incineration. The NO<sub>x</sub> found was 30 µg/liter of effluent gases or 10.8 ppm by volume.

## 2. Results of Incineration Testing at Rocky Mountain Arsenal.<sup>3</sup>

The subject test to be outlined here, resulting from the test plan<sup>4</sup> "Determination of Feasibility of Incinerating Monoethanolamine (MEA) Waste from Fort McClellan, Alabama," was conducted on 6 December 1974.

The hydrazine furnace scrubber system in the 538 complex was utilized for this test. The test operating conditions are detailed in table F-2. The furnace temperature was maintained at approximately 1800°F utilizing a MEA/HD feed rate of approximately one gallon per minute. Analysis of the ambient air and stack gases using DB-3 bubbler method for HD content was conducted and results were indicated in tables F-3 through F-5. All bubblers indicated a negative HD contamination.

In addition, the scrubber brine was sampled throughout the test for free caustic and HD as shown in table F-6. The average caustic content was approximately 2.7%, and no HD contamination was detected.

With regard to stack emissions, RMA laboratory personnel conducted the SO<sub>2</sub> stack gas analysis utilizing the Dynascience analyzer. The average concentration of SO<sub>2</sub> in the stack was 12 ppm as shown in table F-7. Army Environmental Hygiene Agency (AEHA) personnel conducted the NO<sub>x</sub> stack analysis as well as the determination of particulate emissions. The average NO<sub>x</sub> concentration was approximately 65 ppm, and the particulate emissions were within established standards.

It is concluded that the existing equipment located in the 538 complex will efficiently incinerate the MEA/HD waste. Some additional equipment will be necessary for a full-scale production run, mainly HD transfer and pumping equipment. It is anticipated that in actual production the feed rate would be about two gallons per minute. It should be noted that this incineration system would most economically be operated on a three-shift basis; however, there is considerable latitude in the actual mode of operation. Since the present operational plans call for the phosgene transfer to utilize a separate scrubber system in place of the furnace scrubber system, there would be no physical interference between the two operations. It is doubtful, however, from a manpower standpoint, that the HD incineration could be accomplished prior to the completion of the phosgene transfer that is presently scheduled for 1 September 1976.

## 3. Test Plan - Determination of Feasibility of Incinerating Monoethanolamine (MEA) Waste from Fort McClellan, Alabama.<sup>4</sup>

### a. Objective.

Plans for the demilitarization of eight one-ton containers of distilled mustard (HD) have been made at Fort McClellan, Alabama. The process will involve the chemical neutralization of mustard agent (HD) with the decontaminant monoethanolamine (MEA). Current plans now envision shipment of this waste (approximately two hundred 55-gallon drums) to RMA for incineration.

The objective of this test is to determine if the demilitarization waste from Fort McClellan, Alabama, can be incinerated without excess pollution in the RMA bulk mustard liquid furnace, and if the resulting brine solution is similar to that generated during the mustard demilitarization program.



Table F-2. Test Operating Conditions, 6 December 1974

Time	Hydrazine furnace					Quench/scrubber system								ESP fan	
	Combustion chamber temperature	Furnace temperature	Gas valve extent open	Combustion air damper extent open	Natural gas pressure	Quench inlet temperature	Scrubber inlet temperature	Quench inlet pressure	Quench outlet/scrubber inlet pressure	Scrubber outlet pressure	Scrubber outlet temperature	Precipitation outlet pressure	Inlet pressure	Outlet temperature	
	°F	°F	%	%	psig	°F	°F	inHg	inHg	°F	inHg	inHg	inHg	°F	
hr															
1238	1875	1380	98	37	15	850	152	2.5	3.8	10.8	152	25	25	78	
1258	1490	1575	98	40	15	975	160	2.0	3.5	10.5	160	25	25	80	
1315	1550	1700	98	44	15	1120	165	1.5	3.0	11.0	165	25	25	85	
1330 <sup>a</sup>	1600	1625	98	44	15	1100	165	1.5	3.0	9.9	165	25	25	90	
1345	1650	1900	90	44	10	1280	180	1.5	2.8	10.5	180	25	25	95	
1400 <sup>b</sup>	1575	1750	45	44	8	1175	180	1.5	2.8	10.0	180	25	25	95	
1415	1675	1800	46	44	8	1200	172	1.5	2.8	9.0	172	25	23.5	95	
1430	1675	1850	43	44	8	1200	175	1.2	2.5	7.5	175	22	21.5	140	
1445 <sup>c</sup>	1800	1830	30	44	5.5	1200	175	1.2	2.4	7.0	175	21	20.0	180	
1500	1775	1830	30	44	5.5	1200	175	1.2	2.4	7.0	175	20.5	19.5	195	
1575	1800	1850	30	44	5.5	1175	175	1.2	2.4	7.0	175	20.5	20.0	210	

<sup>a</sup> At 1330 hours, changed from MEA drum to MEA-HD drum run No. 18.<sup>b</sup> At 1405 hours, switched to drum No. 2.<sup>c</sup> At 1445 hours, switched to drum No. 4.

Table F-3. Stack Gas Analysis, 6 December 1974

Sample laboratory No.	Time	H content (DB-3)	Time received in laboratory
	hr	mg/m <sup>3</sup>	hr
72870	1245-1327	<0.03*	1535
72871	1327-1521	<0.03**	1535

\* During MEA-only burn.

\*\* During MEA and H burn.

Table F-4. Hydrazine Area Analysis, 6 December 1974

Sample laboratory No.	Time	H content (DB-3)	Time received in laboratory
	hr	mg/m <sup>3</sup>	hr
72869	1330-1530	<0.004	1535

Table F-5. MEA Fumes Analysis, 6 December 1974

Sample laboratory No.	Time	H content (DB-3)	Time received in laboratory
	hr	mg/m <sup>3</sup>	hr
71764	1038-1053	<0.009*	1100

\* Because of 15 minutes and <0.25 µg/ml, a lowest DET rate, the mg/ml exceeds hydrazine area bubbler. However, no effect from fumes on bubblers was noted.

Table F-6. Brines from Special H Test, 6 December 1974

Sample laboratory No.	Sample No.	Specific gravity	H content (DB-3)	Time	[N <sub>2</sub> O]
			mg/m <sup>3</sup>	hr	μg
71765	0	1.078	<.25	1200	2.88
71766	1	1.080	<.25	1250	2.62
71767	2	1.081	<.25	1300	2.59
71768	3	1.078	<.25	1310	2.66
71769	4	1.082	<.25	1320	2.64
71770	5	1.083	<.25	1330	2.55
71771	6	1.083	<.25	1340	2.62
71772	7	1.083	<.25	1350	2.68
71773	8	1.084	<.25	1400	1.91
71774	9	1.084	<.25	1410	2.42
71775	10	1.085	<.25	1420	2.39
71776	11	1.084	<.25	1430	2.37
71777	12	1.085	<.25	1440	2.22
71778	13	1.092	<.25	1450	2.72
71779	14	1.087	<.25	1500	2.96
71780	15	1.087	<.25	1510	3
71781	16	1.097	<.25	1520	2
71782	17	1.096	<.25	1530	3.06
71783	18	1.098	<.25	1540	3.03
71784	19	1.098	<.25	1550	2.99
71785	20	1.097	<.25	1600	2.81



Table F-7. Tabulation of Stack Analysis for SO<sub>2</sub>, 6 December 1975

Time	Concentration of SO <sub>2</sub>	Remarks
hr	ppm	
0925	0	Calibrate Dynascience at 500 ppm std.
1240	0	Start MEA burn 0.5 gal/min
1310	0	MEA flow 3.0 gal/min
1315	8	
1325	10	
1327	10	Switch to 1st drum MEA/H, 1.5 gal/min
1335	10	
1345	10	
1355	12	
1405	10	Switch to 2d drum
1415	10	
1417	-	ESP on
1425	15	
1440	15	
1445	0	Switch to 3d drum
1500	15	
1515	15	End 3d drum, purge system
1521	70	
1530	12	
1535-1550	-	Zero calibration 500 ppm std - OK

b. Operating Parameters.

The set of parameters of the incineration system for the test MEA waste incineration are:

- (1) Furnace chamber temperature, 1800°F
- (2) Gas valve percent open, as required
- (3) Combustion air damper percent open, as required
- (4) Gas pressure, 5.5 psig
- (5) Quench tower inlet temperature, 1600°F
- (6) Quench tower outlet temperature, 130°F

c. Test Procedure.

(1) The flow rate of the MEA waste will be regulated in such a way as to maintain a furnace temperature of 1800°F. Prior to charging the MEA waste, one drum of MEA will be charged to the furnace to insure all monitors and process equipment are properly operating. When this has been established, charging of the MEA waste will begin. The MEA waste may be charged to the system by the method most convenient to the engineer in charge (i.e., through an unload booth, direct gravity feed into the agent holding tank, direct feed into line, etc.) – the only prerequisite is that steady flow can be maintained. The waste is nontoxic, however, it is irritating to mucous membranes. Handlers should wear gloves, aprons, and an aspirator mask if direct contact is involved. If the liquid contacts the skin, it should be washed off as soon as possible.

(2) Due to the volume of waste available (69 to 110 gallons), only one parameter may be explored to assure steady state furnace and detection equipment operation. This is in the best interests of obtaining reliable results.

(3) Air sampling will be accomplished by the following methods:

- SO<sub>2</sub>: Dynascience sampler connected to electrostatic precipitator (ESP) stack
- NO<sub>x</sub>: Teco and PDS method tests in ESP stack
- NO<sub>2</sub>: By using results of NO<sub>x</sub> tests
- Opacity: By trained and certified observers

Organizations responsible for supplying operating personnel for the detection equipment will be as follows:

- SO<sub>2</sub>: RMA
- NO<sub>x</sub>: Army Environmental Hygiene Agency (setup aid from RMA)
- NO<sub>2</sub>: Covered by NO<sub>x</sub>
- Opacity: Army Environmental Hygiene Agency

(4) Liquid sampling of the scrubber brine will be made before the start of the incineration and at ten-minute intervals throughout the duration of the incineration. These will be subsequently analyzed by RMA to determine percent of salt in the solution and the composition of these salts.

d. Test Variables.

(1) All strip charts from air sampling operations will be clearly marked in relation to the time taken. All charts will be zeroed prior to operation startup.

(2) All brine samples will be clearly marked as to the sampling time.

e. Documentation.

(1) Applicable standard operating procedures (SOPs) will be utilized during the entire incineration test. The alarms normally utilized in plant operations will be employed and applicable procedures followed if an alarm condition occurs.

(2) A report of these tests will be published in letter form stating the results and distribution made. Brine samples analyzed at RMA will be compared to previously analyzed brines from the mustard demilitarization operation.

4. Results of Air Pollution Source Survey at Rocky Mountain Arsenal by the US Army Environmental Hygiene Agency.<sup>5</sup>

a. Purpose.

Personnel of the Air Pollution Engineering Division, US Army Environmental Hygiene Agency, participated in testing of the hydrazine furnace and air pollution control system at Rocky Mountain Arsenal (RMA), Denver, Colorado, on 6 December 1974. The purpose was to monitor the oxides of nitrogen and visible emissions resulting from the incineration of reaction products of monoethanolamine (MEA) and distilled mustard.

b. Background.

Personnel of the Disposal Engineering Division, Manufacturing Technology Directorate, Edgewood Arsenal, are designing a suitable method for disposing of mustard currently stored at Fort McClellan. This method involves detoxifying the liquid mustard by reaction with MEA in the ratio of five parts of MEA to one part of mustard. (Stoichiometric neutralization of the mustard requires at least three parts of MEA for each part of mustard.) After detoxifying the mustard at Fort McClellan, it is planned to ship the reaction products to the hydrazine furnace complex at RMA. The test plan<sup>4</sup> in the previous section was written to study the feasibility of incinerating this MEA-mustard waste. This division was requested to support this study. Emission testing performed by RMA personnel involved measurement of the following emissions:

- (1) Any active mustard, utilizing bubblers for sampling
- (2) Any active mustard, utilizing a Tracor continuous gas chromatograph monitor
- (3) SO<sub>2</sub>, utilizing a Dynascience continuous SO<sub>2</sub> monitor.



c. Findings and Discussion.

Data from the oxides of nitrogen measurements are summarized in tables F-8 and F-9. The data were obtained in accordance with method 7 of the Federal Register.<sup>6</sup> Three samples were taken while the system was burning MEA only, and the remaining six samples were drawn when the detoxified waste was being burned. Results of these measurements indicate that nitrogen oxide emissions will not be excessive during the incineration of MEA or the detoxified waste.

The visible emissions were studied first without the electrostatic precipitator and finally with two chambers operating. The unit failed to meet the 20% equivalent opacity standard<sup>7</sup> without the precipitator. However, it did meet the opacity standard with the precipitator operating. The emissions were white particulates that might have been salt carried with the gases leaving the packed bed scrubber.

The test plan had designated the feed rate at two to three gallons per minute, but this flow rate could not be achieved because of the use of a small rotameter in the waste feed line. As a result, the feed rate for this test was reduced to approximately one gallon per minute.

There was an insufficient amount of detoxified waste available for this test to allow proper quantitative measurement of particulate emissions. However, based on past evaluations of the controls of this incinerator as well as observations during this test, it can be assumed that applicable particulate emission standards could be met. To insure this, particulate emission testing should be performed at the outset of subsequent incineration of this waste. Doing so would also allow proper determination of optimum waste feed rates, which can be expected to be greater than the approximate one gallon per minute achieved during this test.

Table F-8. Furnace Warmup – Nitrogen Oxides Data, 6 December 1974

Graduated flask number	Time	Feed to furnace	Intermittent furnace temperature	NO <sub>x</sub> concentration
	hr		°F	ppm
1	1245	MEA <sup>a,b</sup>	1380	15
2	1250	MEA	c	19
3	1255	MEA	1575	21

<sup>a</sup>Monoethanolamine was fed to the furnace prior to test to bring chamber temperature up to 1800°F.

<sup>b</sup>Flow rate was one gallon per minute.

<sup>c</sup>Not specifically noted, but known to be greater than 1380°F and less than 1575°F.

Table F-9. Actual Test — Nitrogen Oxides Data, 6 December 1974

Graduated flask number	Time	Feed to furnace <sup>a</sup>	Intermittent furnace temperature	NO <sub>x</sub> concentration
	hr		°F	ppm
4	1335	MEA-mustard waste	1625	77
5	1340	MEA-mustard waste	b	60
6	1345	MEA-mustard waste	1900	65
7	1350	MEA-mustard waste	c	70
8	1355	MEA-mustard waste	2000	68
9	1400	MEA-mustard waste	1760	90

<sup>a</sup>Flow rate was 0.94 gallon per minute.

<sup>b</sup>Not specifically noted, but within the range of 1625° to 1900°F.

<sup>c</sup>Not specifically noted, but within the range of 1900° to 2000°F.

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## APPENDIX G

### MONOETHANOLAMINE - SUPERTROPICAL BLEACH (STB) COMPATIBILITY

The inclosure herein describes the compatibility of various concentrations of monoethanolamine (MEA) and supertropical bleach (STB). It also considers a small side study of the effect of MEA on brazing materials.

#### REPORT OF DECONTAMINATION TESTS<sup>1</sup>

In accordance with the discussion of 9 September 1974, a number of tests were conducted to determine the compatibility of MEA with dry, powdered STB, solutions of STB in water, and commercial bleach (Clorox). The results of these tests are summarized in table G-1.

As indicated, the addition of MEA to dry, powdered STB resulted in a strongly exothermic reaction and, in one case, produced ignition of the MEA additive. The addition of 1:1 or 2:1 ratios of MEA to 50% w/w solution of STB in water also produced a vigorous exothermic reaction. The addition of 1:1 and 10:1 ratios of MEA to 5% w/w solutions of STB in water produced a temperature rise of approximately 13°C. The addition of a 1:10 ratio of MEA to 5% w/w solution of STB produced a temperature rise of approximately 4°C. The addition of MEA to a 5% commercial bleach solution in a 1:1 ratio resulted in a 16°C temperature rise.

As requested, six-inch lengths of copper and silver brazing rod that were provided were tested by exposure to a 5:1 MEA/H decontamination reaction solution over a period of several days. Observations made over this period are shown in table G-2.

In general, the copper and silver brazing rods showed some slight attack by the MEA/H solution. However, no serious deterioration of either wire was observed during the 148-hour test period.

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<sup>1</sup>Pistritto, J. V. Report of Decontamination Tests. Chemical Laboratory, Edgewood Arsenal. 17 October 1974.

Table G-1. Addition of Monoethanolamine (MEA) to Dry and Aqueous Supertropical Bleach (STB) and Commercial Bleach (Clorox)

Amount	Test material	Amount of MEA	Observations
		ml	
10 gm*	STB, dry	20*	Continued addition of STB to MEA resulted in production of a tarry residue and reddish flame in approximately 3 minutes.
5 gm*	STB, dry	20* (with 1 ml of H <sub>2</sub> O added)	Great release of heat resulting in the production of smoke or fumes on addition of STB to MEA solution.
10 gm	STB, dry	1	Large amount of smoke or fumes produced after 3 minutes from the addition of the MEA to the STB powder. A charred residue was produced. MEA or H <sub>2</sub> O vapor condensed on walls of glass container above STB powder.
12 ml	50% STB (by weight) in water	12	The slurry temperature increased from 25° to 115°C after addition of the MEA to the STB slurry. Extensive bubbling occurred and the slurry turned light brown in color.
12 ml	50% STB (by weight) in water	24	The slurry temperature increased from 25° to 115°C after addition of the MEA to the STB slurry. Considerable bubbling occurred and the slurry turned light brown in color.
5 ml	5% STB (by weight) in water	5	Addition of MEA to STB solution produced some temperature rise (13°C).
1 ml	5% STB (by weight) in water	10	Addition of MEA to STB solution produced some temperature rise (12°C).
10 ml	5% STB (by weight) in water	1	Little if any heat produced (4°C rise).
5 ml	Commercial bleach	5	Addition of MEA to bleach resulted in a solution temperature rise of 16°C (27° to 43°C).

\* Approximately.

Table G-2. Exposure of Copper and Silver Brazing Rod to a 5:1 Solution of Monoethanolamine (MEA)/Mustard (H)\*

Observation number	Time	Observation	
		Copper rod	Silver rod
	hr		
1	3	Some evidence of corrosion at liquid-air interface.	No evidence of reaction.
2	5	Same as 3-hour observation.	Same as 3-hour observation.
3	72	Reddish coating and blue-black spots on wire surface above liquid, wire surface below liquid showed loss of lustre.	Wire above liquid had reddish coating, wire below surface generally had a black coating except for one small area of reddish coating.
4	148	Wire rinsed with water. Wire surface below liquid had an irregular brown-black coating – no evidence of extensive corrosion. Wire surface above liquid had a few brown-black spots on surface – no evidence of extensive corrosion.	Wire rinsed with water. Wire below surface had an irregular black coating – no evidence of extensive corrosion. Wire above surface had a light reddish-brown irregular coating – no evidence of extensive corrosion.

\*Copper and silver brazing rods tested in individual MEA/H solutions to avoid corrosive effects of galvanic action.



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